12-2011

An Evaluation of Disturbance-Induced Nutrient Changes and Climate Responses of Loblolly Pine Xylem

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To the Graduate Council:

I am submitting herewith a dissertation written by Rebecca Lynne Stratton entitled "An Evaluation of Disturbance-Induced Nutrient Changes and Climate Responses of Loblolly Pine Xylem." I have examined the final electronic copy of this dissertation for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, with a major in Natural Resources.

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An Evaluation of Disturbance-Induced Nutrient Changes and Climate Responses of Loblolly Pine Xylem

A Dissertation Presented for
the Doctor of Philosophy
Degree
The University of Tennessee, Knoxville

Rebecca Lynne Stratton
December 2011

DEDICATION

For all the poor trees I've burned and cut down in the name of science –
it's been fun and I've learned lots, but you won't be the last…

ACKNOWLEDGEMENTS

This dissertation would not have been possible without the help and support of my advisor, committee members, colleagues, family, and friends. I want to thank my advisor Dr. Wayne Clatterbuck for his guidance, support, and patience. I also want to thank my committee members Dr. Henri Grissino-Mayer, Dr. Tom Waldrop, Dr. Jennifer Franklin, and Dr. Amy Johnson for their support and advice.

It was a pleasure working with the U.S. Forest Service Center for Forest Disturbance Science and the University of Tennessee Laboratory of Tree-Ring Science. I am grateful for all of the help I received in harvesting, processing, measuring, and analyzing samples. My field work would not have been possible without the assistance of Dr. Waldrop, Helen Mohr and the Center for Forest Disturbance Science field crew, Chuck Flint, Gregg Chapman, Mitch Smith, and Eddie Gambrell. I am grateful for the financial support to run my nutrient analyses provided by the Center for Forest Disturbance Science. Many thanks to Dr. Grissino-Mayer and Grant Harley for their expertise and assistance in developing the loblolly pine chronology and climate analyses. I am eternally grateful to Dr. Clatterbuck and the Department of Wildlife and Fisheries for their financial support through Graduate Student Research Assistantships to allow me to follow my passion for forest ecology research.

Finally, I want to thank my family and friends for their love and support. Thank you to Mom and Dad, for your love, encouragement, and all the care packages. Thank you to Misty Huddleston, Joel Putnam, and Grant Harley for helping me, supporting me, and keeping me grounded throughout my research and dissertation. Thank you to Gordon McSpadden, for your love, support, patience, and encouragement.

ABSTRACT

Dendrochronological techniques are currently limited to the identification of visible fire scars. However, through the development of new dendrochemical techniques, the potential exists to provide insight into a broader array of pyric ecosystems. In addition, the ability to identify historic climate-growth responses provides a better understanding of the conditions under which historic fire regimes occurred.

This study provides the groundwork for the identification of a dendrochemical nutrient fire signature in xylem and identifies the climate-radial growth responses of loblolly pine (*Pinus taeda* L.) on five sites in the Piedmont of South Carolina. Changes in N, P, K, Ca, Mg, Zn, Cu, Mn, Fe, S, and Na concentrations in xylem as a result of a single fire were examined. The influences of monthly mean temperature, monthly total precipitation, Palmer Drought Severity Index (PDSI), Palmer Hydrological Drought Index (PHDI), and selected ocean-atmospheric oscillations such as El Nino-Southern Oscillation (ENSO), North Atlantic Oscillation (NAO), Pacific Decadal Oscillation (PDO), and Southern Oscillation Index (SOI) on radial growth using univariate and multivariate techniques were also assessed.

Soil and duff analyses indicated sites were similar, but nutrient poor. Xylem mobile and immobile nutrient concentration analyses differed significantly through time for N, P, K, Ca, Mg, Zn, Cu, Mn, and S. No interactions were significant, thus identification of a nutrient fire signature was unsuccessful. The lack of significant interactions is attributed to the methodology used to collect the xylem nutrient samples; however, immobile nutrients indicate the most potential for future research.

Climate-tree growth analyses indicate climate plays a role in the growth of mature loblolly pine in the South Carolina Piedmont. Temperature, precipitation, PDSI, positive phase NAO, and positive phase PDO are the primary drivers of loblolly pine radial growth. Temporal consistency results suggest drought sensitivity increases with tree age. Unlike the climate variables, the relationships between ocean-atmospheric oscillations and radial growth appear to be more cyclical in nature and only influence growth in positive phases.

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CHAPTER I
INTRODUCTION TO FIRE ECOLOGY, DENDROCHEMISTRY, AND DENDROCLIMATOLOGY

Influence of Fire on Ecosystems

Fire alters ecosystem dynamics via effects on structure, function, and composition of vegetation. Fire effects are defined as the physical, chemical, and biological impacts of fire on an ecosystem (DeBano et al., 1998). Direct and indirect effects of fire on trees vary depending on many factors, including fuel loading, arrangement and composition, and tree health, vigor and available resources. These and other environmental factors associated with fire regimes impact fire effects and fire behavior. Healthy, vigorously growing trees with ample available resources are less likely to succumb and die from the detrimental effects of fire than stressed or resource limited trees. Stress and associated stress hormones, as a result of fine root death, cambial death, crown scorch, and disruption of water and resource movement within a tree, can negatively impact a tree. Indirect impacts on trees, such as changes in soil, hydrology, and species composition, can be beneficial or detrimental depending on the ecosystem and species.

Fuel Chemistry

Combustion is a physical-chemical process in which molecules are broken down. DeBano et al. (1998) suggest it is much like a reverse of photosynthesis. In addition to the polymeric organic compounds, macronutrients and micronutrients are present in plant material. These nutrients are required for plant growth and health. A review of fuel chemistry studies by Johnson and Miyanishi (2001) across ecosystem and fuel types found that nutrients, with the exception of nitrogen and sulfur, have high volatilization thresholds and tend to be released as inorganic ions or some level of decomposed molecule, depending on combustion efficiency. Because these cations have high temperature thresholds and are not easily volatilized, large quantities are available on the soil surface after fire and in ash.

Disturbances and Nutrient Cycling

Disturbances, such as forest management practices and natural disturbance regimes, have varying effects on short-term and long-term nutrient cycling. Forest management, with the exception of whole tree harvesting, tends to have little long-term effects on nutrient cycling as long as the system does not change land uses (Johnson, 1992). Unlike the relatively short-term effects of traditional harvesting, whole tree harvesting (harvesting of bole, limbs, and leaves or needles) results in long-term nutrient deficiencies (Boyle et al., 1973; Johnson, 1992). Other forms of forest management such as fertilization increase the relative size of the nutrient pools and uptake, but if not implemented correctly can increase leaching (Vose, 1988).

Natural disturbances can have a wide range of influences on nutrient cycling depending on the scale and impact of the disturbance. Disturbances in loblolly pine ecosystems, such as ice, insect, and wind events, tend to have minor influences on nutrient cycling. These low severity disturbances create a short-term shift in nutrient cycling, but do not tend to significantly impact sizes of nutrient pools. Fire is the only natural disturbance in loblolly pine ecosystems that can significantly alter nutrient cycling. Loblolly pine stands that experience low to moderate severity fire do not usually decrease the total site nutrient pool, but some losses occur through oxidation, volatilization, ash transport, leaching, and erosion (Raison et al., 1985). Less common, high severity fires can have more substantial losses and alter the total size of nutrient pools. Although fire does not tend to alter the total size of nutrient pools, the relationship between nutrient availability and site fertility are not tightly linked; as a result the instantaneous increase in nutrient availability does not always translate to increased tree growth (Clayton, 1976; Schoch and Binkley, 1986).

Dendrochemistry

Dendrochemistry, or the science of interpreting elemental analysis of tree rings, can be used to identify temporal biochemical changes that occur within a tree as a result of environmental changes. Dendrochemical techniques have primarily been used in environmental monitoring of heavy metals contamination, fertilization, and acid rain (Bondietti et al., 2009; Guyette et al., 1991; Smith et al., 2009). A few studies have also evaluated dendrochemical analyses for natural disturbances, such as volcanic activity, and how changes in nutrient cycling alter the climate-growth response (Pearson et al., 2009; Kern et al., 2009; Ünlü et al., 2009). Kern et al (2009) reported that increased sulfur deposition resulting from anthropogenic activities degraded the relationship between temperature and tree growth responses. Similarly, Pearson et al. (2009) reported increases in sulfur, calcium, and other elements in wood and associated increases in tree growth following a volcanic eruption.

Natural disturbances, such as hurricanes, floods, and fire, can have varied effects on nutrient cycling, which theoretically can be quantified via dendrochemical analyses. Hurricanes shift carbon and nitrogen cycles because significant portions of the aboveground biomass are killed and converted to woody debris, causing nutrient pools to increase (Scatena et al., 1996). Flooding has been shown to alter nutrient composition in xylem (St. George et al., 2006). In the Southeast, fire tends to have more significant impacts to nutrient cycling than other natural disturbances because fire frequency is higher than the return interval of most other natural disturbances. However, no dendrochemical elemental analysis studies associated with fire have been conducted.

Dendroclimatology

Climate plays an important role in multiple physical, chemical, and biological processes in the environment (Cole and Rapp, 1981). The amount and type of precipitation influences weathering, leaching, decomposition rates, and plant uptake of water and nutrients. Temperature and its associated seasonality influences decomposition rates, species presence, and

physiological responses of vegetation, including dormancy, transpiration, and respiration. Boreal ecosystems, for example, tend to have longer turnover rates with less litterfall, yet have larger forest floor accumulations of organic matter than temperate and tropical ecosystems due to temperature effects (Marschner and Rengel, 2007).

Dendroclimatological research uses tree rings to study climate-growth relationships and climate patterns from recorded history to reconstruct past climate. In addition, the examination of tree growth-climate relationships is used to analyze radial growth responses to climate variables and to determine limiting environmental factors. Fluctuations in nutrient cycling have been shown to alter climate sensitivity (Kern et al., 2009). When nutrients are the primary limiting factor influencing growth, changes in nutrient availability will influence the magnitude of the potential growth response to climate. Because changes in climate can alter physical, chemical, and biological ecosystem processes, it is therefore plausible to also identify changes in nutrient cycling as a result of climate change and vice versa.

Current Dendrochemical and Dendroclimatological Situation of Loblolly Pine

The role of fire's effects on nutrient cycling and tree uptake and storage is essential for understanding fire-adapted ecosystem dynamics. Studies in agricultural systems suggest fire releases nutrients bound in the crop residue and the organic layer of the soil; however, few studies have been conducted regarding remobilization of nutrients released from fire (Marschner and Rengel, 2007). In addition, little is known about the role of macronutrient and micronutrients on productivity of natural ecosystems in relation to fire effects (DeBano et al., 1998).

Presently, fire history knowledge is based on dendrochronological techniques (identification and dating of fire scars), sediment records (e.g. charcoal), documented fires, or witness trees. Although these proxy records can provide annual resolution and seasonality of fires, limitations exist for all of the techniques (Swetnam et al., 1999). The primary limitation in using these techniques is that the fire intensity and severity must be high enough to scar trees or leave a charcoal deposit. However, many ecosystems, particularly in the southeastern United States, are adapted to low intensity and low severity fires which do not leave fire scars and rarely experience high intensity fires. The use of charcoal deposits is also limited to areas in which charcoal can accumulate, such as in a lake, wetland, or in the soil, and is also influenced by the fire size, severity, and intensity (Whitlock and Anderson, 2003). This constraint severely limits the applicability of these techniques in determining fire regimes and potential management implications.

Fire-Nutrient Signature Hypothesis

The premise of a fire-nutrient signature is based on the potential for changes in stored nutrient concentrations to form when new cells are initialized in the cambium. Because individual trees can respond differently to fire-induced nutrient changes, finding a nutrient, or suite of nutrients, that are most responsive to fire is of interest. Changes in stored plant-immobile nutrients, especially Ca, Fe, Zn, Mn, and Cu, are likely the best candidates for a fire-nutrient signature

because they do not tend to translocate. These nutrients are required for basic life functions of a tree; therefore, significant deficiencies or excesses of these nutrients should be evident in the xylem if significant changes occurred as new xylem was formed. Although not of primary interest for identifying a fire-nutrient signature, N, K, P, Mg, S, and Na were also analyzed to identify whether or not patterns exist in the mobile nutrients similar to the immobile nutrients. Climate also plays a role in nutrient cycling and will also be examined to identify tree radial growth responses and their relation to nutrient changes.

Purpose of this Research

The purpose of this study was to examine the role of fire and climate on within-tree nutrient cycling and tree radial growth responses for loblolly pine growth in the South Carolina Piedmont ecosystems. Nutrient cycling and the influence of fire on nutrient cycles and forest ecosystems are discussed. The relationship between nutrient concentrations of xylem in burned and unburned stands is examined, as well as determining if changes in nutrient concentrations associated with fire can be detected. Establishing a fire signature to detect nutrient changes as a result of fire in xylem provides an extension of fire history and reconstruction capabilities beyond the presence of a known fire-scar to any forested ecosystem with a history of fire. The influence of climate is also examined to identify climate-growth relationships and examine the relationship between nutrients, climate, and tree radial growth responses.

Specific research objectives and hypotheses include:

1. Fire signature

- Test the feasibility of using inductively coupled plasma mass spectrometry (ICP-MS) for identifying mean differences in nutrient concentration as a result of fire in loblolly pine xylem.
    - H_0: Nutrient concentration resolution using ICP-MS can be used to identify differences in nutrient composition of loblolly pine xylem.
    - H_A: Nutrient concentration resolution using ICP-MS cannot be used to identify differences in nutrient composition of loblolly pine xylem.
- Identify mean differences in nutrient concentration for loblolly pine xylem in burned and unburned stands.
    - H_0: No differences occur in nutrient concentration of loblolly pine xylem in burned and unburned stands.
    - H_A: Nutrient composition means differ in loblolly pine xylem in burned and unburned stands.
- Determine if a nutrient fire signature relationship exists for loblolly pine burned under low severity in the Piedmont region of South Carolina.
    - H_0: No nutrient fire signature exists in the nutrient composition of loblolly pine xylem.
    - H_A: A nutrient fire signature exists in the nutrient composition of loblolly pine xylem.

2. Climate-growth relationship

- Identify the climate variables that influence diameter growth in loblolly pine plantations in the Piedmont region of South Carolina.
    - H_0: No relationship exists between climate variables, such as temperature or precipitation, and annual tree growth.
    - H_A: Climate variables are related to annual tree growth.
- Determine if the climate-tree growth relationship is consistent over time.
    - H_0: The climate-tree growth relationship does not change over time.
    - H_A: The climate-tree growth relationship changes over time.
- Determine if changes in nutrient concentrations alter the climate-tree growth relationship.
    - H_0: Changes in nutrient concentrations do not alter the climate-tree growth relationship.
    - H_A: Changes in nutrient concentrations do alter the climate-tree growth relationship.

CHAPTER II
LITERATURE REVIEW OF NUTRIENTS IN FOREST ECOSYSTEMS: FOCUS ON FIRE ECOLOGY IN SOUTHEASTERN PINE FORESTS

Nutrients in Forested Systems

Site characteristics provide the basis for all nutrient cycling. The geology, soils, topography, slope, aspect, elevation, latitude, amount of organics, atmospheric deposition, and past land use influence the cycling potential (Cole and Rapp, 1981). Soils and the underlying geology define the physical, chemical, and biological properties within which nutrient cycling occurs. Soil type and parent material dictate porosity, bulk density, cation exchange capacity (CEC), and soil organisms, among other properties.

Topographical factors (including slope, aspect, and elevation) influence soil depth, temperature, decomposition rate, and moisture regime (Cole and Rapp, 1981). Slope affects soil depth. As the degree of a slope increases, the soil depth tends to decrease. Aspect of the site influences moisture regimes with southern and western aspects tending to be drier than northern and eastern aspects. Elevation and latitude influence temperature and decomposition rates; increased elevation and latitude decreases temperature and decomposition rates.

Vegetation on a site influences the biological processes and the rates of the chemical processes. Different species of vegetation have different nutrient requirements. The soil type, forest type, age, stage of development, and presence, abundance and diversity of vegetation affect relative size of nutrient pools, or total amount of nutrients present, and the flux of nutrients within the ecosystem (Cole and Rapp, 1981). In most forested systems, cycling of nutrients shifts between the overstory trees and the understory during stand development. The stage at which a forest reaches crown closure, for example, can be a critical point in the nutrient cycling of many forest ecosystems because prior to crown closure, the understory plays a more prominent role in the nutrient cycles (Cole and Rapp, 1981). Loblolly pine forests have a predictable change in the relationship of nutrient needs and rates of turnover as a function of stand age and development (Switzer and Nelson, 1972). Similarly, other forested systems experience a shift from understory to overstory dominance of nutrient cycling during stand development until the overstory begins to senesce, at which point the role of the understory begins to increase (Cole and Rapp, 1981; Kazimirov and Morozova, 1973; Turner, 1975).

Unmanaged forest ecosystems with low frequency and low severity disturbance regimes tend to have more predictable nutrient pools than do managed forests or forests with higher frequency and/or higher severity disturbance regimes. Influxes and effluxes of nutrients occur as a result of disturbances. Conifer systems respond to changes in the nutrient pools and tend to have more efficient nutrient use than deciduous systems, primarily because conifer systems do not lose their leaves annually.

A review by Cole and Rapp (1981) of numerous studies across all forested ecosystems reported deciduous systems tend to have 50 percent greater nitrogen requirements than conifer systems at the same level of production, and suggested that conifer systems appear to meet all of their

annual nitrogen requirements, approximately 39 kg/ha, through uptake. Additionally, the review indicated deciduous systems meet their nitrogen requirements through uptake and translocation from older tissue. As a result, nitrogen requirements tend to be strongly correlated to growth rates for conifer systems, but correlations for deciduous systems are weak to moderate.

Role of Nutrients in Tree Growth and Development

Macronutrients and micronutrients are essential for plant growth and development. Each of these nutrient types plays an essential role in the physiological needs of flora. Macronutrients tend to be essential for structural or functional components of cells. Micronutrients tend to be catalysts or components of enzymes that aid in growth or development, but their specific roles are less understood than macronutrients and require more in-depth research for loblolly pine. The distinction between macronutrients and micronutrients is related to plant needs rather than uptake amounts.

Soil Nutrients

There are 13 essential soil-supplied nutrients (Table 1). The primary macronutrients required in large quantities for plant growth and survival are: nitrogen (N), potassium (K), phosphorus (P), calcium (Ca), sulfur (S), and magnesium (Mg). Macronutrients are required in larger quantities than micronutrients, ranging from 0.1 to 5 percent, or 100 to 5000 parts per million (ppm), of dry plant tissue. The micronutrients essential for plant growth in small quantities are: boron (B), iron (Fe), zinc (Zn), manganese (Mn), copper (Cu), chloride (Cl), and molybdenum (Mo). Most micronutrients generally comprise less than 0.025, or 250 ppm, of dry plant tissue.

Table 1. The 13 essential soil-supplied nutrients, including plant and soil mobility, and primary plant function.

Nutrient	Plant Mobile	Soil Mobile	Primary Function in Plants*
Macronutrients			
N	Mobile	Mobile	Proteins, enzymes, hormones, metabolic processes
P	Mobile	Immobile	All aspects of tree physiology
K	Mobile	Intermediate	Enzymes, plant water potential
Ca	Immobile	Mobile	Cell wall structure, middle lamella, nutrient uptake, neutralizing base
S	Mobile	Mobile	Proteins, enzymes, amino acids, vitamins, chlorophyll production, root growth, seed production
Mg	Mobile	Immobile	Chlorophyll, growth enzyme activation and function, ATP production, phosphate transfer, translocation and metabolism of carbohydrates
Micronutrients			
B	Immobile	Mobile	Cell wall formation catalyst, nutrient uptake and transfer, nutritional sugars transfer and production, hormone movement, pollination, seed development
Fe	Immobile	Immobile	Chlorophyll formation catalyst, O^2 transport, enzyme function and synthesis
Zn	Mobile	Mobile	Enzymes, enzyme catalyst, function and synthesis of auxin, growth hormones, and proteins, carbohydrate transport, metabolism, and regulation
Mn	Immobile	Immobile	Activator component of enzymes in respiration, nitrogen metabolism, and photosynthesis, chlorophyll formation, sugar metabolism, catalyst in oxidation and reduction reactions
Cu	Immobile	Mobile	Growth regulator, component of several enzymes, aids in nitrogen metabolism
Cl	Mobile	Mobile	O^2 production in photosynthesis, ion charge balancing, turgor regulation, plant metabolism, stomata regulation, K^+ balance
Mo	Mobile	Intermediate	Enzyme function in nitrogen activation, structural component of nitrate reductase, increases nitrogen fixation in leguminous trees, seed development

*Primary function in plants from Brady and Weil, 1999; Havlin et al., 2004; Kabata-Pendias and Pendias, 1992; White and Broadley, 2001.

Fire Effects on Soil and Organic Matter

Fire effects on soil and organic matter properties and processes vary depending on ecosystem, fire intensity, and fire severity (Hungerford et al., 1990). Soil physical structure is not generally affected under low and moderate severity fires in the Southeast (Certini, 2005). The combustion of organic matter and humus creates an instantaneous increase in the litter layer decomposition rate and mobilizes nutrients and resources, which can be beneficial to surviving biota.

Influence of Fire on Nutrient Fates

Fire has significant contributions to nutrient cycles (Johnson et al., 2004). Fire can affect the complex and interrelated chemical and biological processes within an ecosystem. Chemical processes and reactions that can be altered include nutrients with low volatilization temperatures, pH, cations, and cation exchange capacity. Soil organisms and biologically mediated processes, such as decomposition, are sensitive to ecosystem changes.

Nitrogen tends to be the most limiting nutrient in most forest ecosystems and is the only soil nutrient not supplied to the soil by chemical weathering of parent material. Soil heating and combustion of organic matter volatize N. Because N has the lowest volatilization temperature of any plant nutrient at 200 °C, N volatilization is the primary process responsible for N losses during a wildland fire (White et al., 1973). When N is volatized during wildland fire situations, total N pools decrease, but available N tends to increase for a short period. Wildland fire-induced losses of N vary by ecosystem, but complete loss of N generally occurs when organic matter is completely consumed and temperatures reach in excess of 500 °C (DeBano et al., 1998). However, complete combustion of organic matter and N volatilization do not occur in most loblolly pine wildland fire situations. Low productivity ecosystems tend to have more significant decreases in productivity as a result of fire-induced N losses than high productivity ecosystems (Nave et al. 2011). In low productivity ecosystems, biological processes play a significant role in nutrient cycling.

In addition to N, S and P have volatilization thresholds that can be reached in wildland fire situations. Sulfur has a volatilization temperature of 375 °C (Tiedemann, 1987). Unlike N, S cannot be fixed by biological processes and must be added to ecosystems via atmospheric deposition. Phosphorus has a volatilization temperature of approximately 774 °C (Raison et al., 1985). Although uncommon in southeastern ecosystems, under complete organic matter combustion, approximately 60 percent of total P is lost (DeBano et al., 1998). Other nutrients generally have volatilization temperatures greater than that reached in wildland fire situations. Combustion of organic matter generally increases availability of most soil nutrients; however, combustion also increases the potential for nutrient losses via erosion, runoff, particulate loss in smoke, leaching, or immobility due to incomplete combustion (DeBano and Conrad, 1976; Raison et al., 1993; Grier, 1975; Boerner, 1982).

Low Intensity Fire Effects on Soil Nutrient Cycling

Soil heating will transform nutrients from organic to inorganic forms, and modify the decomposition process, thus increasing nutrient availability. Low intensity fires only consume the litter layer and may lightly char the duff layer, but the mineral soil chemistry is not altered. Nutrient cycling is influenced in that nutrients typically bound in the litter and duff layers are released faster than they are typically degraded by soil processes and organisms increasing the nutrient concentrations in the soil solution, the duff, and in the soil organic matter.

The relationship between available nutrients and nutrient pools is complex. Available nutrients and nutrient pools within a forested ecosystem can shift as a result of fire. Although soil nutrient pools may decrease as a result of fire, nutrient availability often increases for a short period because the soil and organic matter properties and processes are altered (Wright and Bailey, 1982). Soil nutrient pools decrease as a result of fire through some combination of combustion, pyrolosis, volatilization, ash transport, leaching, and erosion. Nutrient availability increases due to the instantaneous release of the nutrients from organic matter as a result of combustion or pyrolosis, combined with an increase in soil pH and CEC. However, nutrient pools are also subsequently decreased as a result of volatilization, ash transport, leaching, and erosion. The effects of fire on ecosystem nutrient pools and fluxes are primarily dependent on the severity of the fire. Available soil nutrients are a product of physical, chemical, and biological properties of soils (Neary et al., 1999). Low intensity and severity fires can increase available soil nutrients and promote herbaceous flora, while high intensity and severity fires can destroy organic matter and soil structure leading to leaching, erosion, and changes in soil properties.

Sizes of Nutrient Pools

The sizes of nutrient pools and available nutrients varies by stand, forest, and ecosystem within the Southeast. Nitrogen and carbon (C) cycles are the two primary nutrient cycles of interest to natural resource managers, ecologists, and economists because N and C cycles dictate ecosystem budgetary constraints and their cycles are closely tied. These budgetary constraints limit the rates of the biological processes and other nutrient fluxes within ecosystems (Gorham et al., 1979). Fire has significant contributions to the global C and N cycles (Johnson et al., 2004). Historically, fire was much more prevalent on the landscape and forested ecosystems did not store as much carbon as today's forests (Houghton et al., 2000). Nitrogen is the most limiting nutrient in most forest ecosystems and the C:N ratio influences nutrient cycling (DeBano et al., 1998). The effects of fire on ecosystem nutrient pools and fluxes are primarily dependent on the severity of the fire.

A nationwide study of forested ecosystems by Houghton et al. (2000) reported that since European settlement and the exclusion of fire, ecosystems have become carbon sinks and total carbon stored has increased. Current live undisturbed biomass in the United States averages approximately 140 t C ha^{-1}, while ecosystems that are repeatedly burned on 2-year cycle average approximately 4 Mg t C ha^{-1}. Although a 2-year fire interval is not common in most ecosystems, shifts in the disturbance regimes have significantly changed the carbon cycle in most forested

ecosystems. A landscape level regional study of the southeastern Coastal Plain suggests forested lands accumulate approximately 1 t C ha^{-1} yr^{-1} and although disturbance causes changes in the size of the nutrient pools, many are short-term and have little impact on the net landscape exchange of carbon (Binford et al., 2006).

A study by Boerner et al. (2008) across multiple ecosystem types estimated the amount of carbon stored within ecosystems was 45 percent stored in vegetation (50 to 174 Mg C ha^{-1}), 38 percent stored in soil organic matter (40 to 120 Mg C ha^{-1}), 10 percent stored in the forest floor, and 7 percent stored in dead wood (combined forest floor and dead wood 4 to 151 Mg C ha^{-1}). Low severity fire was not found to have significant changes to the stored C in eastern ecosystems. Total stored C was approximately 196 to 210 Mg C ha^{-1} and fire only reduced the stored C by 1 to 7 Mg C ha^{-1}. Fire in Nevada *Pinus jeffreyii* forests consumed approximately equal percentages of C and N (9 to 12 percent) but tended to consume greater amounts of above ground N (71 percent) than C (21 percent; Johnson et al., 2004). Losses of N from fire were compared to the equivalent of more than 1,000 years of atmospheric N deposition or approximately 10,000 years of N leaching at current rates. However, C losses will be replenished with regrowth of vegetation.

Stored and used C and N vary for different ecosystems. Within a stand, the nitrogen pool and available nitrogen will vary not only by species, but Samuelson (2000) reports differences will occur by family within a single species and allocation of the nitrogen will also vary within the trees. Natural, unfertilized stands of loblolly pine in the upper Coastal Plain of South Carolina had an average N soil availability of approximately 0.73 to 2.5 Mg N ha^{-1} with forest floor N availability averaging 13.4 to 56.2 Mg N ha^{-1} (Birk and Vitousek, 1986). Although carbon and nitrogen decreased with decreasing nitrogen availability, sites with the lowest N had the highest nitrogen use efficiency. Annual carbon use in young loblolly pine stands in North Carolina was approximately 7.5 Mg C ha^{-1} with the total ecosystem carbon pool ranging from 12.8 to 20.2 Mg C ha^{-1} yr^{-1} (Maier et al., 2004).

Low severity fires tend to have little impact on soil C but impact increases with increasing severity (Johnson, 1992). Fires cause a reduction in the O horizon with no change or increase to mineral soil C when fire severity and intensity is low, but high severity and intensity fires completely remove the O horizon and can severely burn the soil surface and char to a depth of 10 cm (Johnson, 1992; DeBano et al., 1998). The forest floor of a low severity periodic fire regime in a South Carolina forest regained organic matter that approached control levels between fires, and organic matter and N increased to the amount lost by fire suggesting no long-term effects on the ecosystem (Wells, 1971). A review of low severity fire studies reported increases in soil C due to incorporation of charcoal and partially burned organic matter into mineral soil and an increase of N-fixing species (Johnson, 1992). High severity fires tended to have significant soil C and N losses of 40 to 50 percent throughout the soil profile with up to 100 percent of surface organic matter consumed.

Litter is one of the primary fuels in southeastern forests and as a result, some of the most significant changes occur to the litter in the event of a fire. Deciduous forests produce more litter by weight, volume, and quantity because they lose their leaves annually. Coniferous trees keep their needles for three to five years. The long, thin shape and composition of southern coniferous

pine (*Pinus* spp.) needles are highly flammable because they dry faster and have a much higher proportion of energy-dense lignin and lipid content than deciduous leaves. Different *Pinus* species produce differing amounts of litter and decomposition rates vary. *Pinus echinata*, for example, produces greater increases of mineral soil organic matter input yet has lower accumulation of litter than loblolly pine (Gilmore and Rolfe, 1980). Loblolly pine accumulates approximately 44 g C m^{-2} yr^{-1} of litter (Schlesinger and Lichter, 2001; Schiffman and Johnson, 1989). The average weight of the litter layer in young and 40-year old loblolly pine in South Carolina and Georgia, respectively, was approximately 8875 kg ha^{-1} (Kodama and Van Lear, 1980; Brender and Cooper, 1968). Decomposing loblolly pine litter has been found to be a net sink for P and N while a net source for potassium (K), magnesium (Mg), and calcium (Ca) with variable decomposition rates depending on site and climate (Sanchez, 2001). A study that evaluated the reduction of litter by low intensity fire in a loblolly pine forest in South Carolina reported that litter dry weight of 890 g m^{-2} was reduced by 33 percent to 596 g m^{-2} (Lewis, 1974). Low severity and intensity fires tend to only consume part of the litter layer and some of the duff, but high severity and intensity fires can remove all of litter and organic matter (Certini, 2005). Although most nutrients have high volatilization temperatures, consumption of the litter and organic layers can release previously bound macronutrients and micronutrients making them more readily available, which often results in losses via ash transport, runoff, or leaching.

The influence of fire on soils tends to be minimal in southeastern ecosystems because fire intensity and severity tend to be low. Soil physical structure is not generally affected under low and moderate severity fires. Soil heating is a concern with high fuel loads because soil heating can kill soil organisms and have detrimental effects on the soil physical and chemical properties. Wildland fire ranges in temperature from 300 °C to 1400 °C and the degree of soil heating is dependent on the fuel type, rate of fire spread, and the residence time of the fuel (DeBano et al., 1998). Soil organisms, including bacteria, plant roots, fungi, seeds, small mammals, and macro- and micro-invertebrates, generally have a threshold temperature tolerance of 50 °C to 120 °C before death occurs. The residence time of the heat also influences tolerance (DeBano et al., 1998). Although uncommon in the Southeast, high severity and intensity fires (fires that consume all of the litter and duff) can degrade soil structure, porosity, decrease water retention, and kill soil organisms due to soil heating (Certini, 2005). Water repellency increases and hydrophobic layers can form under high severity fires in addition to irreversible decreases in porosity, water retention, and elasticity. These changes in the soil structure decrease the infiltration rate and make soils more erodible (DeBano et al., 1998). The temperature at which soil structure is irreversibly changed varies by soil texture and structure. Soils with high clay content can tolerate relatively high temperatures with loss of OH groups occurring at 460 °C, and irreversible changes in water of hydration occurring at 980 °C (DeBano et al., 1977).

Flux of Nutrients within the Ecosystem

Nutrient cycling that results from ecosystem processes tends to fluctuate the relative sizes of nutrient pools. Nutrient fluxes within ecosystems tend to shift the distribution of nutrient concentrations to other ecosystem components, but can also result in fluxes out of the ecosystem. Disturbances, including fire, can change nutrient cycling and nutrient fluxes within a forested ecosystem, including litterfall, decomposition, mineralization, and plant uptake.

Litterfall

Litterfall in forested *Pinus* spp. ecosystems averages approximately 125 g m^{-2} (Harrington and Edwards, 1999). Loblolly pine forests treated with prescribed fire tend to have consistently lower average litterfall and lower mass than unburned stands (Renschin et al., 2002). Nutrient analyses also show increased concentrations of K, Ca, and Mg but lower concentrations of C in litterfall under the burn treatment than in the unburned controls. Litterfall tends to be lower in burned systems when fire severity is low and fire does not scorch the crown because trees tend to retain their needles and allocate nutrients to other areas that were damaged by the fire. Under moderate and high severity fires, litterfall is greater than unburned stands because needles are killed and subsequently fall prematurely.

Decomposition

Fire alters decomposition rates of organic matter and fuels in forested ecosystems. Nutrient changes that normally take place over a period of many years via microbial decomposition can occur within a few seconds during fire (DeBano et al., 1998). Rates of decomposition vary by fuel type, with fine litter decomposing rapidly to larger woody debris taking decades to breakdown. Decomposition rates of litter in loblolly pine systems in the absence of fire average 311 g C m^{-2} yr^{-1} (Kinerson et al., 1977). King et al. (1997) recorded decomposition rates of roots ranging from 2 to over 20 years. Decomposition rates fluctuate with regard to available moisture and stand age and development (Maier and Kress, 2000). In a mature loblolly pine prescribed burn study, decomposition rates and the concentration of N released from the forest floor increased (Schoch and Binkley, 1986). Increased decomposition rates result in increased availability of nutrients and pH (Certini, 2005). With increased pH, more nutrients also become available in the typically acidic soils of pine ecosystems.

Mineralization

Mineralization rates in forested ecosystems differ by soil type, vegetation type, and season. As stands develop, they have increased nitrogen demands but the soil supply of nitrogen becomes limited due to continual immobilization (Miller et al., 1979). Rates of mineralization have been shown to increase with increased nutrient availability via fertilization and changes in vegetation (Gurlevik et al., 2004; Wood et al., 1992). Coastal Plain loblolly stands that have not been burned tend to have C:N ratios from 25:1 to 36:1, but burned C:N ratios range from 43:1 to 58:1 (Binkley et al., 1992; Bell and Binkley, 1989).

The effect of fire on mineralization appears to have a temporal influence. Although fire initially increases available N that results in the "green-up" effect immediately post fire, mineralization rates decrease post-green-up and immobilization of N becomes greater which reduces available N. A review of low severity studies showed N-fixing plant species increase post fire which increases immobilization and decreases the mineralization rate (Johnson, 1992). Nitrogen poor loblolly pine sites in South Carolina had mineralization rates of 210 mg N m^{-2} on unburned sites but mineralization rates decreased with burning to 78 to 87 mg N m^{-2} (Bell and Binkley, 1989). Although burn treatment forest floor and mineral soil C:N ratios were higher than the control treatment, greater than 95 percent of the N was immobilized, which decreased N availability.

Annual burning on a different loblolly pine site in South Carolina showed increases of N because of increased activity of N-fixers but mineralization rates were low (Wells, 1971).

Plant Uptake

The nutrient requirements and uptake of plants varies by species. For all species, specific minimum amounts of nutrients and water are required for basic physiological functions and growth. Within species, genetics plays a role in nutrient use and efficiency. Nitrogen efficiency is a trait in which genetics plays a role because some families are better adapted to grow in lower N available soils than others (Li et al., 1991). Nutrient concentration and ratios in the soil solution also influence plant uptake. In nitrogen limited loblolly pine systems, the efficiency of N increases the growth per unit of N absorbed and allows the trees adapted to the N limited sites to have better productivity (Birk and Vitousek, 1986). Damage to roots and stems from fire can cause plants to decrease nutrient uptake ability and productivity; however, most fire-adapted species are able to recover quickly.

The amount of N available for plant uptake is determined by the microbial release of N in the form of mineralization and the incorporation of N into the biomass via immobilization (Binkley and Hart, 1989). The effect of fire on forested ecosystems tends to decrease total nitrogen but increase available nitrogen (DeBano et al., 1998). In low severity fires, little to no N tends to be lost from the forest floor and losses that occur are balanced by mineral N gains due to increased nitrate from microbial activity (Mroz et al., 1980; Wells, 1971; Lewis, 1974). However, high severity fires can volatize significant amounts of nitrogen and thus significant N losses occur. After fire, concentrations of ammonium nitrogen increase as a result of volatilization in the soil, which enhances plant growth and causes the "green-up" effect (DeBano et al., 1998). In addition to ammonium, nitrite, nitrate, and other cations are released and made more readily available to plants if not lost to leaching. In systems that do experience nutrient losses after high to moderate severity fires, plants come in and recolonize and most properties can be recovered (Certini, 2005).

Carbon is required in nearly all aspects of plant physiology, is the basic building block of cells, and is the basis for tree growth. Carbon uptake for eastern forests is estimated at 5 to 12 Mg C ha^{-1} (Boerner et al., 2008). Although carbon is an integral part of the nutrient cycle and tree growth, it is not a limiting nutrient in forested ecosystems because C is in ample supply in the atmosphere. Fire tends to increase labile C pools and shift the C:N cycle to a higher carbon to nitrogen ratio, which can limit nutrient uptake. After the initial flush of N from ammonium nitrogen, soils balance and nitrogen becomes limited again because available C is greater than pre-fire concentrations. Fire also converts organic C into CO_2 via thermal degradation of organic molecules (DeBano et al., 1998).

Flux of Nutrients Out of the System

Nutrient fluxes out of ecosystems occur as a result of some ecosystem processes and disturbances. Because fire changes the nutrient cycling and decomposition rates, the rate of nutrient fluxes out of the system also changes. Nutrients flux out of the system primarily through volatilization, denitrification, leaching, and erosion.

Volatilization

Fire begins to volatize N at 200 °C, which is below the temperature of combustion for most forest fuels (DeBano et al., 1998). Once combustion occurs, N is volatized at a directly proportional rate to the amount of organic matter combusted. Volatized organic N is converted to gaseous forms and the organic matter is decomposed via pyrolosis and combustion (Pyne, 1984; DeBano et al., 1998). Once temperatures reach 500 °C, over 50 percent of the N and organic matter can be volatized (Neary et al., 1999). In addition to nitrogen, phosphorus (P >774 °C), potassium (K >760 °C), sulfur (S >800 °C), sodium (Na > 880 °C) and calcium (Ca >1240 °C) are frequently lost into the atmosphere during combustion as particulates (Weast, 1988). Nutrient losses by ash convection ranged from 3.6 lbs/ac for P to 23.1 lbs/ac for N (Johnson, 1992).

Although the amount of volatized N varies, significant proportions of the volatized N do not leave the site and are trapped on leaves, branches, and stems which are then rinsed back into the soil. A study of nutrient fluxes in a South Carolina loblolly pine forest reported increased nitrate after the first rainfall after a prescribed fire but not after subsequent rains, suggesting the nitrate was transported via stemflow and collected by rain drops and returned to the forest floor (Lewis, 1974). Not all nitrogen that is volatized in the soil leaves the burn area. Nitrogen that is volatized in the soil tends to be translocated through the soil profile rather than lost to the air (DeBano et al., 1998).

Denitrification

Denitrification does not play a significant role in most southeastern N cycling because anaerobic conditions are not common in forested systems. Annual losses due to denitrification are estimated from 1 to 3 kg N ha^{-1} yr^{-1} in UK forests (Dutch and Ineson, 1990). Loblolly pine forests are typically on sandy soils with adequate drainage. Areas that do not have adequate drainage likely have higher denitrification rates but these areas are also not likely to be harvested or treated with fire. Little research has been done on denitrification as a result of fire due to the overwhelming losses of N to volatilization (DeBano et al. 1998). Denitrification rates have been found to increase when plant material or herbicides are applied, suggesting denitrification rates would increase as a result of fire (Vitousek and Matson, 1985). Fire would therefore have the most significant effect on denitrification rates if the burned area was flooded immediately after a fire occurred. Most prescribed fires are also conducted in the dry season which would also suggest denitrification rates would be low or change little.

Leaching

Leaching and erosion can result in significant fluxes of nutrients out of an ecosystem. Upper soil layers and organic matter are efficient at adsorption of cations but movement occurs in runoff and wind-blown particulate matter (Lewis, 1974). Erosion is not typically problematic on undisturbed forested sites because plant roots tend to hold soil, even on steep slopes in the South. Disturbances such as fire that breakdown organic matter and destroy plant roots can result in significant losses of soil and nutrients from erosion processes, particularly on steep slopes.

Nutrient leaching from the forest floor and soil in disturbed ecosystems is more problematic across all sites because these losses are related to soil and organic matter quantity and nutrient holding capacities. Soils of the Southeast tend to have low soil nutrient holding capacity and low cation exchange capacity because they are sandy and porous which allows nutrients to quickly leach out of the system. Organic matter in the upper part of the A horizon can provide over 50 percent of the cation exchange capacity in some forest soils (DeBano et al., 1998). Natural loblolly pine stands from South Carolina to Louisiana generally have a CEC ranging from 10 to 40 mmol kg^{-1} and occur on a variety of soil types (Birk and Vitousek, 1986; Sword et al., 1998). Typical leaching rates in East Tennessee loblolly pine stands are approximately 2 to 14 $kmol_c$ ha^{-1} yr^{-1} (Johnson and Todd, 1987; Richter et al., 1994).

Fire can have significant effects on leaching rates. Severity of fire is the most influential to leaching rates because increasing severity increases the amount of organic matter that is consumed which then increases the probability of leaching. Microbial immobilization is one of the most important processes to prevent N leaching (Bell and Binkley, 1989). However, microbial growth and survival are highly dependent on the amount of soil heating. Fire increases pH which increases cation solubility. In low severity burned South Carolina pine forests, fire increased the solubility of cations and within 30 days, 80 mm of rainfall leached 70 percent of the total leachable nutrients and reduced the ion yield from 45 to 83 percent (Lewis, 1974). To minimize leaching and erosion, fire severity should be minimized and post-fire precipitation should be low, particularly on steep slopes.

CHAPTER III
DENDROCHEMISTRY AND DENDROCLIMATOLOGY BACKGROUND

Dendrochemistry

Analytical dendrochemistry methods can be used to identify the chemical composition of trees for a wide range of environmental changes. There are numerous analytical instruments and each are designed to address specific qualitative or quantitative sample needs from solids to gases, atomic to molecular scales, and can range from non-destructive to destructive sampling. Each instrument uses a stimulus, usually in the form of electromagnetic, electrical, mechanical or nuclear energy, to elicit a response (Skoog et al. 2006). A detector is then used to identify, record, or indicate a change in the response and displayed on a readout devise.

Commonly Used Techniques in Dendrochemistry - Mass Spectrometry and Optical Atomic Spectroscopy

Mass spectrometry is a versatile tool used for identifying elemental composition and concentration, and can be used for nearly all of the elements in the periodic table. There are four basic steps in a mass spectroscopic analysis: atomization, conversion of a fraction of the atoms in the sample to a stream of ions, separating the ions based on their mass-to-charge ratio, and counting the number of ions by type based on the sample ionized (Skoog et al. 2006). There are numerous mass spectroscopy techniques based on how the sample is atomized and the mass analyzer.

Inductively coupled plasma mass spectrometry (ICP-MS) is widely used and useful in dendrochemistry because it has low detection limits (0.02 to 0.1 ppb), multielement capabilities, high speed, and easily interpretable spectra. It incorporates high-temperature argon plasma with a quadrupole mass analyzer to measure the elemental composition and concentration (Adams et al., 1988). The primary disadvantages are that the instrument costs are higher than optical atomic spectroscopy, frequent calibration is required, and sample destruction (Skoog et al., 2006).

According to Skoog et al. (2006), optical atomic spectrometry is similar to mass spectrometry in that it is used to identify elemental composition, but it cannot be used to identify the mass of a sample. Unlike mass spectrometry, optical atomic spectrometry uses an energy source to excite atoms to an energy level which produces a photon (a particle of light). The wavelength of light emitted can then be used to relate to the well-defined energy of an atom.

Laser ablation methods, such as laser induced breakdown spectroscopy (LIBS), use a high energy laser pulse to excite a sample, from which, a spectrograph and detector are used to identify the elemental composition of a sample (Lee et al., 2004). LIBS tend to be minimally- to non-destructive, and can be used with solid samples such as wood, but cannot quantify concentrations without calibration with either wet chemistry or quantitative techniques. The technique was successfully used in a criminal case to link wood from a crime scene to the

suspect, with 99.99% confidence that the spectra of the trace elements in the wood were identical based on the chemical signature (Page, 2005).

Dendroclimatology

Few dendroclimatic studies have been conducted on the climate of the South Carolina Piedmont region and fewer on plantation loblolly pine. Dendroclimatological studies conducted in the southeastern United States have suggested radial growth responses vary by geographic location and site characteristics, but southern yellow pines tend to be most responsive to temperature, precipitation, and the Palmer Drought Severity Index (PDSI). A review of Cook et al. (1998) reported the climate response of loblolly pine in the western part of the Southeast has been linked with drought sensitivity to June rainfall and maximum temperatures and may be drought sensitive at the end of the growing season following a June drought. These drought sensitivity relationships diminish for easterly locations. Weak, non-significant temperature responses were suggested during the dormant season and spring, but the investigators did not suggest further analysis.

A study comparing tree-ring chronologies to climate conducted in southeastern Coastal Plain longleaf pine (*Pinus palustris* Mill.) reported PDSI had the highest correlations with growth, particularly between July and November (Henderson and Grissino-Mayer, 2009). Climate-radial growth responses of loblolly pine in northern Georgia found late growing season temperatures and increased precipitation had positive effects on tree growth (Grissino-Mayer et al., 1989). Virginia pine (*Pinus virginiana* Mill.) and pitch pine (*Pinus rigida* Mill.) in the Virginia Piedmont both responded to temperatures in late fall and precipitation from prior summer (Copenheaver et al., 2002). Shortleaf pine (*Pinus echinata* Mill.) growth and density responses to temperature and precipitation in the South Carolina Piedmont reported strong relationships between early and late growing season temperatures and dormant season precipitation, but no other weather or ocean-atmospheric oscillation climate data were evaluated (Cleaveland, 1975).

Climate

Palmer Drought Indices

A series of drought indices have been developed based on W.C. Palmer's 1965 index to measure the departure of moisture supply. Palmer Drought Severity Index (PDSI) is a meteorological drought index that responds to abnormally wet or dry changes in weather conditions and is calculated based on precipitation, temperature, and available water content of the soil (Palmer, 1965). PDSI was originally derived using an algorithm calibrated for relatively homogeneous regions to produce a standardized index that could be used to compare between locations and months the duration and intensity of long-term drought-inducing circulation patterns (Palmer, 1965; Alley, 1984). The Palmer Hydrological Drought Index (PHDI) was developed to measure cumulative long-term hydrological impacts of drought, which take longer to develop and recover from changing conditions. In 1989, the Palmer Modified Drought Index (PMDI) was developed and differs from PDSI in how the transitional periods between wet and dry spells are calculated (Heddinghaus and Sabol, 1991).

North Atlantic Oscillation (NAO)

Rogers (1984) reported NAO is a monthly index of standardized sea level pressure anomalies in the north Atlantic. The oscillation is a large scale shift in the subtropical high and the polar low pressure centers in the North Atlantic. NAO drives winter climate variability in the North Atlantic region, including North America, Europe, and northern Asia. Although the NAO index varies annually, the oscillation tends to remain in phase for intervals of several years. Positive phase of NAO occurs when there is a strengthening of the Icelandic low and Azores high. The increase in the pressure gradient over the North Atlantic causes cold air to drain off the continent preventing cold air from moving south. The positive phase of NAO primarily influences South Carolina winters by decreasing the potential for wintery weather. The negative phase of NAO occurs when there is a weakening of the two pressure centers allowing cold air to move south. The South Carolina weather resulting from the negative phase of NAO has increased potential for wintery weather and coastal storm tracks.

Atlantic Multidecadal Oscillation (AMO)

According to Kaplan et al. (1998), AMO is an index of North Atlantic sea surface temperature variability between the equator and Greenland. It consists of a series of 20 to 40 year fluctuations in North Atlantic sea surface temperatures. NAO primarily affects air temperatures and rainfall over North America and Europe and is associated with drought frequency and increased frequency of severe Atlantic hurricanes. The warm phase of AMO results in decreased rainfall, while the cool phase of AMO results in increased rainfall and droughts in South Carolina.

El Nino Southern Oscillation (ENSO)

Troup (1965) reported ENSO is derived from monthly values of sea surface temperature differentials between Tahiti and Darwin, Australia from the Southern Oscillation Index. Although the oscillation is identified by changes in sea surface temperatures, sub-surface temperatures respond before sea surface temperatures. These changes in sea surface temperatures affect low-level atmospheric winds, convection, the jet stream, tropical activity, and winter temperatures and precipitation. The warm phase (El Niño) results in increased precipitation and cooler than normal temperatures in South Carolina; while the cool phase (La Niña) results in decreased precipitation, warmer than average winter temperatures, and more favorable hurricane development in the Caribbean and Atlantic.

Southern Oscillation Index (SOI)

According to Troup (1965), the SOI is calculated from the monthly fluctuations in the air pressure difference between Tahiti and Darwin, Australia. It is the atmospheric counterpart to ENSO, and drives the shift in ocean temperatures. The warm phase (El Niño) is characterized by low pressure at Tahiti and high pressure at Darwin, while in the cool phase (La Niña) the reverse pressure gradient occurs. The coupled interaction between the ENSO and SOI result in short-term (2 to 5 year) variations in global climate.

Pacific Decadal Oscillation (PDO)

Mantua (2009) reported PDO is a monthly index derived from sea surface temperatures in the North Pacific Ocean, poleward of 20 °N latitude. The oscillation is similar to ENSO in character, but the phases are longer (20 to 30 years; Zhang et al., 1997). The shifting of Pacific sea surface temperatures alters upper level atmospheric winds and as a result affects Pacific and Atlantic hurricane activity, droughts and flooding, and global land temperatures. The warm phase of PDO is associated with above average sea surface temperatures off the coast of North America from Alaska to the equator creating a horseshoe shape of warmer-than-average temperatures around a core of cooler-than-average temperatures. Warm phase PDO results in below average winter temperatures and above average winter precipitation in South Carolina. The cool phase of PDO is the reverse phenomenon, and results in above average winter temperatures and below average winter precipitation in South Carolina.

CHAPTER IV
STUDY AREA AND SITE DESCRIPTIONS

Study Area History

Prior to 1930, the land around what is now Clemson University was intensively row-cropped. The farming practices decreased soil productivity to submarginal conditions with extensive erosion (Dunn and Holladay, 1977). The foundations of the Clemson Experimental Forest ("Clemson Forest") were a product of improvements to the degraded farmlands and were funded by the Roosevelt Administration's New Deal Programs. In 1934, the Works Progress Administration reclaimed the land by planting trees, stabilizing eroded gullies, and constructing fire towers and recreation facilities. By the late 1930s, Clemson College began supervision of the Forest. In the mid-1940s, silvicultural practices were implemented to manage the forest. The Clemson Forest was established in 1954; and now consists of 7,080 ha of intensively managed forest supporting multiple uses, including sustained timber yield, wildlife habitat, recreational opportunities, and teaching and research programs.

Geology

The Clemson Forest is located in the Piedmont physiographic province of the Appalachian Highlands division (Horton and Dicken, 2001). The Piedmont province ranges from New Jersey to Alabama and is bounded by the Blue Ridge province to the west and the Coastal Plain to the east (Fenneman, 1916; U.S. Geological Survey, 2004). This region is characterized by low, rolling hills with elevations between 50 and 300 m. The study area occurs at elevations ranging from 225 to 290 m. The geology of the Piedmont province is complex and the primary bedrock geology of the study area consists of biotite gneiss and granite gneiss.

Soils

The soils of the Piedmont region are primarily Ultisols formed from felsic, metamorphic, or igneous rocks. Ultisols are geologically old and have had active forces driving soil formation over long periods, resulting in deep, acidic soil profiles with few basic cations (Buol et al., 1997). Ultisols in the United States generally occur in warm humid climates, south of the glacial drift, and have had the pedogenic processes of weathering, leaching, and erosion acting upon them for approximately 70 million years.

The soils present on the study area include the following soil series in decreasing order of prevalence: Madison, Cecil, Pacolet, and Cataula. These soils are classified as Typic and Oxyaquic Kanhapuldults (Web Soil Survey, 2007), and are deep to very deep, moderately to well drained, acidic sandy loams and clay loam soils on slopes ranging from approximately 0 to 20 percent (Soil Survey Staff, 2010). Piedmont Ultisols are weathered and generally lacking in phosphorous. The inherent fertility of the sites is low and the soils are considered nutrient poor.

Climate

The climate of the South Carolina Piedmont region is broadly classified in the humid subtropical zone under the Köppen-Geiger climate classification system (Köppen, 1900; Geiger, 1954; Kottek et al., 2006). This zone is characterized by hot, humid summers and cool winters with relatively even amounts of precipitation occurring in all seasons. The Clemson Forest is located in the NOAA South Carolina Northwest climate division. Temperatures and precipitation vary little across the study area because of similarities in topography, elevation, slope, and proximity (distances between sites range from 2.5 to 15.5 km). Since 1895, mean annual temperatures on the Forest have ranged from 5 °C in January to 26.3 °C in July, with minimum average annual temperatures reaching –1.2 °C and maximum average annual temperatures reaching 32.6 °C (National Climatic Data Center, 2010). Mean annual precipitation on the Clemson Forest averages approximately 137 cm. Precipitation on the Forest is generally in the form of rain; however, periodic ice storms have occurred.

Vegetation

The Clemson Forest has a diverse vegetative community, including pine, hardwood, mixed pine-hardwood stands in various stages of development. The primary forest cover type of interest in this study is classified as eastern forest cover type 81, loblolly pine (Eyre, 1980). Stands were established beginning in the 1930s and were primarily planted to loblolly pine seedlings. All stands were visually estimated to be fully stocked. Intensity of management in the pine stands has varied by stand, and can include periodic thinning and/or burning. Understory vegetation consist of herbaceous vegetation and woody regeneration. Regeneration and midstories are composed of loblolly pine and mixed hardwoods, including oaks (*Quercus* spp.), sweetgum (*Liquidambar styraciflua*), hickories (*Carya* spp.), and maples (*Acer* spp.).

Loblolly Pine

Loblolly pine is one of the most widely studied and commercially important species of the southern yellow pines in the southern United States. It is an adaptable species with a wide range of suitable soil types, climates, and topography (Baker and Langdon, 1990). Loblolly pine accounts for approximately 11.7 million ha across 14 states from southern New Jersey, south to central Florida, and west to eastern Texas (Figure 1).

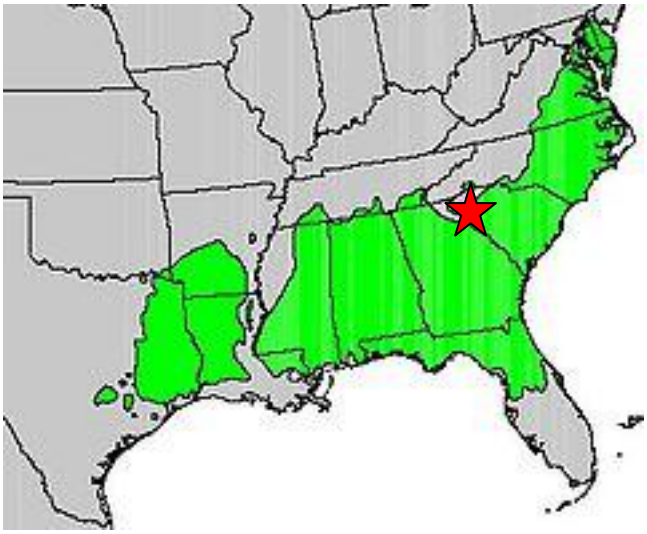

Figure 1. Native range of loblolly pine and study area in Clemson, SC (Little, 1971).

Loblolly pine is a medium-lived species (120 years with a maximum of 275 years) with a high fire resistance in mature trees (Hare, 1965), but low fire resistance in trees under two inches in diameter (Wade, 1993). Loblolly pine is generally the first tree species to colonize following stand level disturbance or old-field succession but frequent disturbance is required to retain the species in future stands (Baker and Langdon, 1990). Growth of loblolly pine is influenced by stand density, competition, environmental factors, and soil physical and chemical properties for natural and managed systems. Prescribed fire is used for site preparation and as an intermediate operation to reduce density, and control competing and understory vegetation (Wade, 1993).

Study Sites

Stands with similar establishment and past management histories were selected on the Clemson Experimental Forest in South Carolina. Stands were 24 to 92 years old with a mean of 46 years and had not been thinned. In most dendrochronological studies, sites are selected based on the Principle of Site Selection, which states trees that are growing in sub-optimal conditions are more likely to be sensitive to climate (Fritts, 1976). In this study, sites were chosen based on similar site characteristics and past management. One fire exclusion stand and four stands with a single known fire were selected for this study. Differing fire years were chosen to ensure any treatment effect detected was a result of fire induced chemical changes and not natural annual variation. All stand characteristics were collected in the field or by using public data. All stands experienced an ice storm in 2006 and the 2002-2003 southern pine beetle attack.

Holly Springs Road

The Holly Springs Road stand (34°44'42.01"N, 82°52'1.88"W) is located on the ridge top and upper side-slopes, northwest of Lake Issaqueena, in the northwestern portion of the Clemson Forest (Figure 2). The elevation measured ranged from approximately 245 to 260 m. The stand is bounded by Lake Issaqueena Road to the south-southeast, a service road to the north and east,

and other stands to the west. The overstory species composition is 45-year old loblolly pine with oaks, hickories, and loblolly pine in the understory and midstory. The understory is a rich mix of woody and herbaceous plants. The site index was 70 feet for loblolly pine at base age 25. The primary soil series is a Madison sandy loam (Web Soil Survey, 2007). The site has had no intermediate operations conducted since establishment with the exception of one prescribed fire in 2001.

East Dam Road

The East Dam Road stand (34°44'38.08"N, 82°50'41.91"W) is located on the ridge top and upper side-slopes with a slight eastern aspect, southeast of Lake Issaqueena, in the north-central portion of the Clemson Experimental Forest (Figure 2). The elevation measured ranged from 250 to 260 m. The stand is bounded by East Dam Road to the east and other stands to the north, south, and west. The overstory species composition is 35-year old loblolly pine with oaks, maples, and loblolly pine in the understory. The understory is sparse with some woody species present. The site index is 70 feet for loblolly pine at base age 25. The primary soil series is a Madison sandy loam (Web Soil Survey, 2007). The site has had no intermediate operations conducted since establishment with the exception of one prescribed fire in 2001.

Watershed Road

The Watershed Road stand (34°37'34.82"N, 82°49'8.23"W) is located on the ridge top in the south-central portion of the Clemson Experimental Forest (Figure 2). The elevation measured ranged from 225 to 235 m. The stand is bounded by Watershed Road to the west, a bench to the east, and other stands to the north and south. The overstory species composition is 55-year old loblolly pine with sweetgum and loblolly pine in the understory. The understory is sparse with some woody and herbaceous species present. The site index is 80 feet for loblolly pine at base age 25. The primary soil series are Cecil sandy loam and Cataula sandy loam (Web Soil Survey, 2007). The site has had no intermediate operations conducted since establishment with the exception of one prescribed fire in 1991.

Swine Center

The Swine Center stand (34°38'45.09"N, 82°48'49.44"W) is located on a flat section of the south-central portion of the Clemson Experimental Forest (Figure 2). The elevation measured ranged from 240 to 250 m. The stand is bounded by Starky Road from the northwest to southeast and other stands to the north, east and west. The overstory species composition is 67-year old loblolly pine with loblolly pine in the understory and midstory. The site index is 80 feet for loblolly pine at base age 25. The primary soil series is a Cecil sandy loam (Web Soil Survey, 2007). The site has had no intermediate operations conducted since establishment with the exception of one prescribed fire in 1991; two prior attempts to burn this stand were conducted in 1988 and 1989, but were not successful.

Outdoor Lab

The Outdoor Lab stand (34°37'39.93"N, 82°50'23.03"W) is located on the ridge top and upper side-slopes in the southwestern portion of the Clemson Experimental Forest (Figure 2). The elevation measured ranged from 225 to 240 m. The stand is bounded by Charlie White Trail from the west and other stands to the north, south and east. The overstory species composition is 45-year old loblolly pine and shortleaf pine to a lesser extent with oaks and maples in the understory and midstory. The understory is sparse with some woody and herbaceous species present. The site index is 70 feet for loblolly pine at base age 25 .The primary soil series is a Pacolet clay loam (Web Soil Survey, 2007). The site has had no fire or intermediate operations conducted since establishment. The stand has a higher hardwood component than that other stands, but all other site characteristics are similar to the fire stands, suggesting the hardwood component is likely the result of lack of fire.

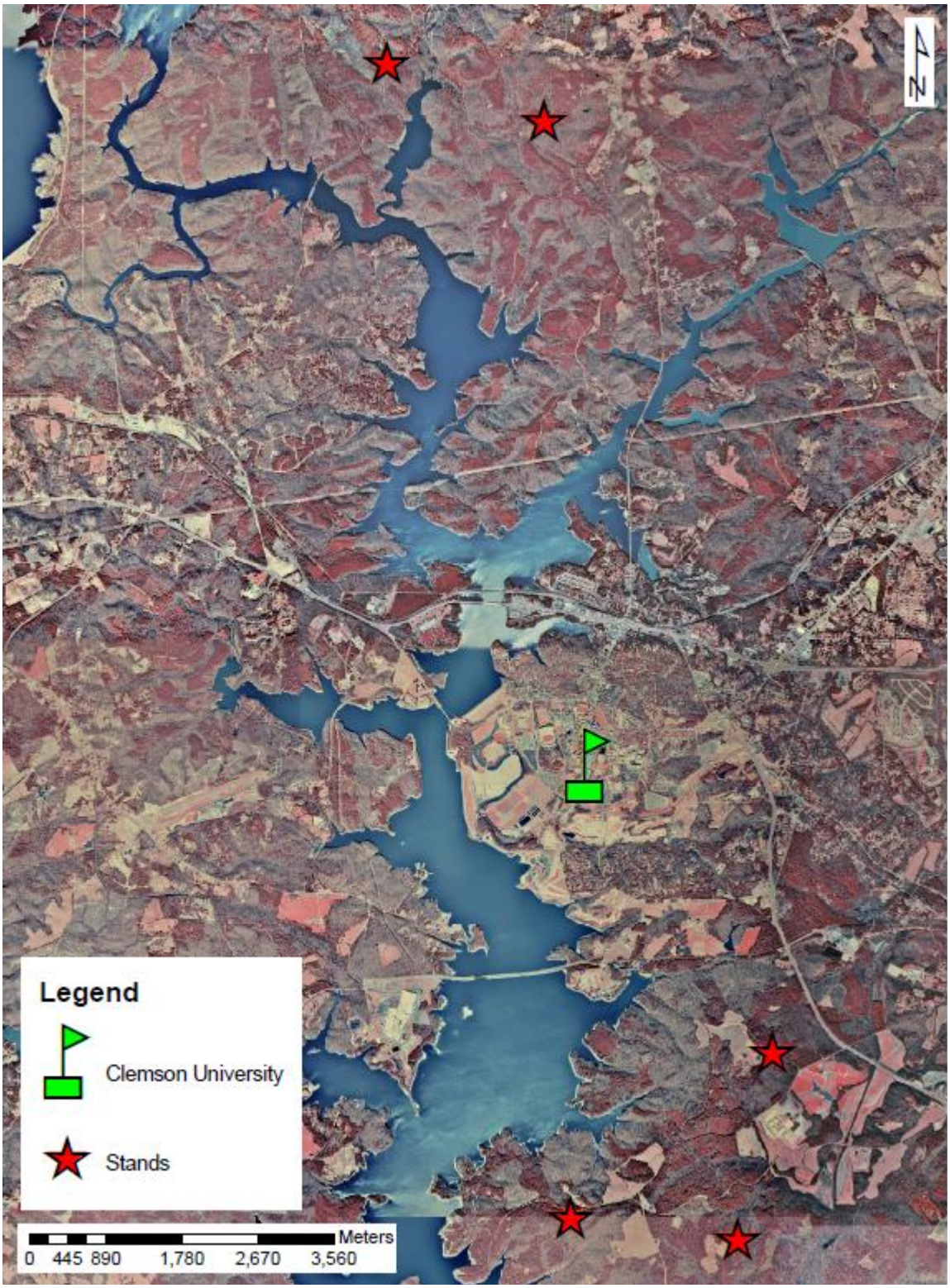

Figure 2. Map of stands, Holly Springs, East Dam Road, Swine Center, Outdoor Lab, and Watershed Road, Clemson Experimental Forest, SC.

CHAPTER V
METHODOLOGY

Field Methodology

At each of the five sites, three loblolly pine trees (15 total trees) were harvested for cross-sections. Tree cores were also collected from a minimum of ten trees from each site (n=68) for chronology development. Each tree was selected using the subjective without preconceived bias approach to be an average dominant or codominant stem and free of visible scars (Mueller-Dombois and Ellenburg, 1974). Prior to harvest or coring, diameter at breast height (DBH), total height, slope position, aspect and evidence of char were documented. The trees that were felled were cut as close to the ground as was deemed safe, approximately 15 cm from the root collar. Cross-sections were removed from the lower 30 cm for analysis. For cored trees, two radii per tree were extracted at approximately 30 cm using Haglof increment borers. Although slopes were minimal across sites, cores were taken at right angles to the slope to avoid potential reaction wood (Grissino-Mayer, 2003). Fifty-three cores were collected across all sites: 13 from Holly Springs, 10 from East Dam Road, 10 from Watershed Road, 10 from Swine Center, and 10 from Outdoor Lab. All samples were stored in ventilated plastic straws and labeled for processing and analysis at the Forest Stand Dynamics Laboratory and the Laboratory of Tree-Ring Science at the University of Tennessee, Knoxville. Because differences in soil organic matter can influence nutrients available to plants, samples of soil, litter, and duff were collected from within 3 m of the bole from 15 to 20 points within the drip line of each tree (n = 15) sampled. Soil was collected from the rooting zone to a depth of 15 to 20 cm.

Laboratory Methodology

Cross-section Preparation

Cross-sections (1 per tree) were dried in a kiln at 43 °C wet bulb/35 °C dry bulb for 72 hours. Once dry, each cross-section was surfaced using a belt sander with progressively-finer sanding paper (ANSI 60, 120, 220, 320, and 400 grit) to prepare the surfaces for dissection and to remove residual material from felling and transportation (Orvis and Grissino-Mayer, 2002). Each cross-section was then scanned using a high resolution scanner at 1200 dpi resolution and imported into WinDendro software. WinDendro is a dendrometric system which utilizes a high resolution scanner and image processing software to aid in dating tree rings. Within WinDendro, each ring was assigned a calendar year with an associated ring width by creating a path from the pith to the outer most and most recent ring. Annual tree rings were visually and statistically crossdated using WinDendro and COFECHA to ensure correct assignment of dates to each tree ring (Holmes, 1983; Grissino-Mayer, 2001). COFECHA uses segmented time series analysis and calculates numerous correlation coefficients to determine if ring-width patterns match across samples (Holmes, 1983; Grissino-Mayer, 2001). Known fire years were identified and marked, then cross-sections were quartered and subsequently cut into approximately 5 × 5 cm radius pieces (henceforth samples) using a band saw.

Core Preparation

Cores were dried for at least 24 hours then mounted upright along the transverse plane on labeled wooden core mounts using Elmer's® glue and masking tape. The mounted cores were prepared for analysis by sanding each core with progressively finer sand paper (ANSI 80, 120, 220, 320, and 400 grit) to smooth the surface and allow for identification of individual cell boundaries (Orvis and Grissino-Mayer, 2002). Each core was then scanned using a high resolution scanner at 1200 dpi resolution and imported into WinDendro. Within WinDendro, each ring was assigned a calendar year with an associated ring width by creating a path from the pith to the outer most and most recent ring. If the outer most ring did not include bark, each ring was assigned a floating date, or a date which is unknown.

Crossdating

The samples and cores from each site were crossdated using visual and statistical crossdating techniques (Yamaguchi, 1991; Grissino-Mayer, 2001; Stokes and Smiley, 1996). Because samples from similar sites generally exhibit similar growth patterns, crossdating can be used to assign a calendar year to each individual ring by matching patterns of narrow and wide rings across samples (Fritts, 1976). Statistical crossdating was primarily used to construct chronologies, but visual and graphical techniques were used to correct misdated and floating samples. Statistical accuracy of the dating was checked using the COFECHA 6.06 software package. To test the correlations between each ring-width series and the average of all the other series in the chronology, 50-year segments lagged by 25 years were used and correlation coefficients below 0.3281 ($P < 0.01$) were flagged. COFECHA provides suggested adjustments to dates if higher correlations could be achieved by shifting the flagged segments by ten years or fewer. For each flagged segment, the correlation coefficients were examined and the specific period that was problematic was determined within the series. The cores were then examined to identify false rings, micro-rings, or breaks in the core that may have led to misdating by using scans of the cores and the cores under a microscope. The years 1970, 1981, and 1988 were the predominant problematic years due to drought and rings were often missing or had micro-rings. Four complacent samples, or samples with little to no variation in ring widths, and cores with extremely low correlation coefficients throughout the series and with no evidence of misdating were removed from the chronology because they did not exhibit a strong regional climate signal.

Xylem Nutrient Analysis Preparation

Using the known dates, three intervals were dissected from all radial slices in each tree. The three intervals (here out called nutrient concentrations or samples) contained annual rings from pre-fire, post-fire, and the fire year. Annual rings from a minimum of one year before and one year after the fire were included to allow for translocation of nutrients. The pre-fire and post-fire samples contained and average of five to seven years of annual rings, dependent on ring-widths and minimum sample weight required for analyses. A minimum of two years was not sampled to serve as a buffer. All annual rings were dissected from the mature (a minimum of 10 years old), sapwood (living xylem) portion of the cross-sections and within the most recent 30 years. No juvenile wood (wood less than 10 years old) or heartwood (dead xylem) was used in the sampling. The three samples dissected from the unburned control were taken from similar dates (1990 to 2002) to the fire-treated intervals of pre-fire, post-fire, and fire year to determine natural

variation within the cross-sections. Approximately three grams of each sample (n = 45) was packaged in paper sample bags provided by the Clemson University Agricultural Service Laboratory. Nutrient content of the whole wood samples were then analyzed using the loblolly pine foliar standard "Orchard Leaf" (N, P, K, Ca, Mg, Zn, Cu, Mn, Fe, S, and Na). Total N concentrations were analyzed using Kjeldahl digestion. Total P, K, Ca, Mg, Zn, Cu, Mn, Fe, S, and Na concentrations were analyzed using inductively coupled plasma mass spectrometry (ICP-MS) analysis via the wet ash HNO_3 plus H_2O_2 technique by the Clemson University Agricultural Service Laboratory. For more information, ICP-MS analysis procedures are detailed at http://www.clemson.edu/public/regulatory/ag_svc_lab/plant_tissue/plant_tissue_index.html. The detection limits of the ICP-MS were within the range of nutrient concentrations in the xylem samples and measurement errors were less than five percent. All results are reported in significant digits.

Xylem Nutrient Statistical Analyses

Using a modified Before-After Control-Impact (BACI) design, nutrient concentration samples were analyzed for the pre-burn, fire year, and post-burn intervals of the fire treatment and the control in SAS. BACI designs are used to evaluate whether or not a change has occurred in an environment (Smith, 2002). The primary drawback of a BACI design is that independence between the periods sampled is required. Because nutrient content of tree rings are inherently autocorrelated, repeated measures were used to remove the dependence between samples and to identify potential translocation within the pre-burn interval. Although foliar nutrient analyses are typically reported in concentrations of N, P, K, Ca, Mg, and S in percent, the nutrient concentrations of xylem were reported using parts per million (ppm) because concentrations are generally one to two orders of magnitude lower than those typically observed in foliar concentrations.

The univariate analyses were generated using SAS/STAT® software, Version 9.1.3 of the SAS System for Windows. Several iterations of analysis of variance (ANOVA) models were examined using standardized nutrient concentrations in combination with potential covariates to minimize sources of variation and increase the signal to noise ratio. The concentrations were standardized by the average number of tree rings per interval to account for concentration differences in individual rings. Because plant-nutrient responses can change with soil pH, pH was used as a potential covariate. Differences between burned and unburned stands (here out called treatments), periods, and interactions for each nutrient were conducted using mixed models analysis of variance, conducted at $\alpha = 0.05$. The Shapiro-Wilk test was used to determine normality and Levene's test was used to determine equal variance. Transformations were used to attain normal distributions and equal variances on P (square root), Ca (square root), Fe (square root), and Na (log). Normality and equal variance could not be attained using transformations for Cu, so analysis of ranks was performed. Least squares means were compared using the Tukey's significant difference test (Quinn and Keough, 2002).

Pearson and Spearman correlation analyses were performed between periods and overall for standardized nutrient concentrations for all immobile and mobile nutrients to identify potential inconsistencies between nutrient relationships. Nonparametric Spearman correlation analyses were used because significant differences occurred between Pearson and Spearman correlation

analyses, suggesting the data were not normally distributed. Correlation analyses indicate linear relationships involving dependence. Correlations in this study are categorized into: low (r = 0.30 to 0.59), moderate (r = 0.60 to 0.79), high (r = 0.80 to 1.0). Differences between interval correlations suggest changes in the ratio relationship and potential causal relationships.

The multivariate analyses were generated using SPSS 16.0 software for Windows. To identify multivariate relationships and interactions among nutrients, multivariate analysis of variance (MANOVA) was performed. Concentration differences among burned and unburned stands, periods, and interactions for each nutrient were conducted using generalized linear models analysis of variance. Because testing multiple comparisons increases the likelihood of family-wise errors, the Bonferroni correction was used with an overall alpha of 0.1 (individual $\alpha = 0.017$ for immobile nutrients only and individual $\alpha = 0.009$ for all nutrients). The Bonferroni correction minimizes family-wise errors by testing each individual hypothesis at a statistical significance of $1/n$ that of a single hypothesis test, so that significance is based on α/n for each hypothesis tested.

Soil and Duff Preparation and Analyses

Soil samples from each tree (n = 15) were packaged in sample bags provided by the Clemson University Agricultural Service Laboratory. Soil nutrient concentrations were analyzed by the Clemson University Agricultural Service Laboratory. Duff samples from each tree (n = 15) were analyzed using the same protocol as xylem samples at the Clemson University Agricultural Service Laboratory. Analysis of variance models were conducted on the soil and duff. Tests for concentration differences among treatments for each nutrient were conducted using mixed models analysis of variance, conducted at $\alpha = 0.05$. The Shapiro-Wilk test was used to determine normality and Levene's test was used to determine equal variance. Transformations were used to attain normal distributions and equal variances on duff Zn (square root) and Mn (square root) and soil Ca (square root) and Mg (square root). Normality and equal variance could not be attained using transformations for soil K, Zn Cu, and Na, so analyses of ranks were performed. Least squares means were compared using the Tukey's significant difference test (Quinn and Keough, 2002).

Chronology Standardization

Standardization of crossdated raw ring width measurements was conducted using the ARSTAN program (Cook, 1985). Raw ring-width measurements were standardized by converting the ring widths into dimensionless indices. Standardizing each series removes individual age-growth trends and autocorrelation, and allows the data to be averaged into a single index chronology for each site (Fritts, 1976). The raw data were conservatively detrended using negative exponential curves and linear regression with a negative slope (Cook and Peters, 1981; Cook et al., 1990; Carrer and Urbinati, 2006). The residual output was then used for climate response analyses to minimize biological year-to-year persistence.

All series were combined to form a master chronology. Descriptive statistics generated by COFECHA and ARSTAN from the master chronology were evaluated for similar average interseries correlation, mean sensitivity, and first-order serial autocorrelation. A high average interseries correlation, a measure of how well samples correlate, was obtained for the master

chronology suggesting accurate crossdating and a common climate signal (Grissino-Mayer, 2001). The mean sensitivity, which reflects year-to-year growth variation, was above average for the Southeast, suggesting the series have considerable variation in ring widths and are likely to exhibit a strong climate response (Holmes, 1983; Grissino-Mayer, 2001). First-order serial autocorrelation was low suggesting minimal biological year-to-year persistence which may obscure the climate signal (Grissino-Mayer, 2001). Combining the individual chronologies allows for the identification of the dominant climate variables to which loblolly pine in the South Carolina Piedmont respond and the changes occurring in climate response over time.

Climate Data Selection

Climate data were obtained from the National Climate Data Center (NCDC) regional weather station data for northwest South Carolina (Division 3802) from 1919 to 2008. The weather data variables selected for analysis were monthly mean temperature, monthly total precipitation, PDSI, and PHDI. In addition to PDSI, PHDI was analyzed because the study area is within close proximity to Lake Issaqueena, and the lake may influence the hydrology of the stands. In addition to weather variables, the North Atlantic Oscillation (NAO), smoothed Atlantic Multidecadal Oscillation (AMO), Southern Oscillation Index (SOI), El Niño-Southern Oscillation 3.4 (ENSO), and Pacific Decadal Oscillation (PDO) data were selected as potential drivers of tree growth.

Climate Analysis

Climate analyses were performed in DendroClim2002, which performs complimentary correlation analysis and response function analysis with bootstrapped confidence intervals to test for variable significance (Biondi, 1997; Biondi and Waikul, 2004). The primary drawback with DendroClim2002 is that the program does not develop a bioclimatic model of tree growth explaining how much variance is explained by the independent climate variables in response function analysis (Fritts et al., 1971; Biondi, 1997). Correlation analyses and response function analyses were performed on each climate variable to determine which climate variables were most significant to tree growth. Once significant climate variables were identified, moving intervals correlation analyses were performed on monthly mean temperature, monthly total precipitation, PDSI, PHDI, ENSO, NAO, PDO, and SOI to determine if tree growth-climate relationships were stable over time.

Correlation Analysis and Response Function Analysis

Correlation and response function analyses were tests used to test the strength of association between the tree-ring chronology and monthly climatic variables (Biondi and Waikul, 2004). Response function analysis is a multivariate test that calculates “response” coefficient estimates using a principal component regression model (Briffa and Cook, 1990; Morzuch and Ruark, 1991; Biondi and Waikul, 2004). Correlation and response function analyses were performed between the tree-ring chronology index and monthly climate variables from September of the previous growing season to December of the current growing season because tree growth is influenced by past and current growing conditions (Fritts, 1976). Coefficients were calculated

over two periods, 1919 to 2008 and 1944 to 2008, to account for differences in insufficient sample depth prior to 1944.

Moving Correlation Analysis

Moving correlation analysis expands on correlation and response function analyses by examining the stability or stationarity of the temporal aspect of the climate-tree growth relationship (Biondi, 1997; Biondi and Waikul, 2004). Similar to correlation and response function analysis, moving correlation analysis provides a sequence of coefficients over time that indicates the magnitude and direction of the relationship; because a moving window is used across the series to calculate coefficients, the analysis provides temporal changes that occurred in the magnitude and direction of the relationship. Although a consistent response to a given climate variable is generally used for reconstructions, inconsistencies in temporal climate-response relationships may be the result of changes in biological responses and processes, including nutrient uptake, storage and availability, or changes in climate.

Moving correlation analyses were performed using DendroClim2002 between tree growth and significant weather and ocean-atmospheric oscillation climate variables from correlation and response function analyses: monthly mean temperature, monthly total precipitation, PDSI, PDHI, NAO, PDO, and SOI. A window length of 32 years, or twice the number of predictor months, was selected (Biondi and Waikul, 2004). Analyses for all climate variables were performed from 1919 to 2008. Analyses start and end dates of ocean-atmospheric oscillation climate variables varied as a result of data availability: NAO from 1951 to 2007, PDO from 1919 to 2001, and SOI from 1952 to 2007. Variables correlated with tree growth are graphically displayed with non-significant intervals shaded green and significant intervals shaded with progressively darker shades of red and blue/purple with increasing significance.

CHAPTER VI
RESULTS FOR FIRE SIGNATURE DETECTION USING ICP-MS IN LOBLOLLY PINE, CLEMSON EXPERIMENTAL FOREST, SC, USA

Correlation Analyses

Results of the correlation analyses suggest the nutrients tend to be moderately to highly correlated with each other (Table 2). Spearman correlations for all periods combined were highly significant for all nutrients. Seven of the eleven correlations between pH and the individual nutrients for all periods were significant, but had low correlations due to multicolinearity resulting from complex pH-nutrient interactions. Although these correlations are statistically significant, the ecological significance is minimal because there is no evident relationship between xylem nutrient concentrations and variations in soil pH in Piedmont loblolly pine ecosystems.

Results of correlation analyses on each of the periods revealed that the relationships between the nutrients were not temporally stable. The relationships between the majority of the nutrients decreased from moderate to strong correlations in the pre-burn period to weak to moderate correlations (r = 0.10 to 0.59) in the fire period then returned to moderate to strong correlations (r = 0.60 to 0.80) by the post-burn period (Tables 3, 4, and 5; Appendix). However, nutrient relationships with P, Zn, Fe, Cu, and Mn did not follow this trend.

Phosphorus relationships with S, Zn, Cu, and Mn did not change between the pre-burn (r = 0.60, 0.76, 0.64, 0.54, respectively) and fire periods (r = 0.54, 0.73, 0.59, 0.53, respectively), while the P:Fe relationship increased in strength (r = 0.24 to 0.75). The post-burn period relationships of P:Zn, P:Mn, and P:Fe (r = 0.67, 0.55, 0.48, respectively) were similar to that of the fire period. The P:S relationship decreased in strength (r = 0.72), while the P:Cu relationship increased in strength (r = 0.89).

Zinc relationships with K did not change between any of the periods (r = 0.71, 0.62, 0.61, respectively). The Zn:Ca relationships did not change between the pre-burn (r = 0.68) and fire periods (r = 0.58), then increased in strength by the post-burn period (r = 0.80). The Zn:Mg relationships were the opposite to that of the overall relationships, in that the strength of the relationship increased then decreased in strength (r = 0.69, 0.85, 0.77, respectively).

Iron relationships with K, Ca, Mg, S, Zn, Cu, Mn, and Na increased in strength from the pre-burn period (r = 0.13, –0.04, 0.09, 0.28, 0.34, 0.34, 0.25, 0.17, respectively) to the fire period (r = 0.63, 0.61, 0.69, 0.41, 0.87, 0.79, 0.64, 0.41, respectively). The Fe:N relationship was the only relationship to not increase in strength from pre-burn (r = 0.31) to the fire period (r = 0.30). The post-burn relationships did not change in strength between Fe and P, K, Ca, Mg, and Mn (r = 0.64, 0.69, 0.65, 0.65, respectively). However, post-burn relationships increased for Fe:N (r = 0.48), Fe:S (r = 0.55), and Fe:Cu (r = 0.88), while Fe:Zn decreased in strength (r = 0.76).

Copper relationships with Zn did not change between the pre-burn (r = 0.79) and fire periods (r = 0.82), then decreased in strength by the post-burn period (r = 0.74). Manganese had similar relationships across all periods for P (r = 0.54, 0.53, 0.55, respectively) and Mg (r = 0.43, 0.50, 0.55, respectively). The Mn:N relationship did not change between pre-burn (r = 0.29) and the fire period (r = 0.17) but was followed by an increase in the strength of the relationship by the post-burn period (r = 0.39). Sulfur manganese relationships decreased from the pre-burn (r = 0.31) to the fire-period (r = –0.12) then increased by the post-burn period (r = 0.27).

Table 2. Spearman correlation coefficients for Piedmont loblolly pine xylem nutrient concentrations across all periods, Clemson Experimental Forest, SC, 2011.

	pH	N*	P*	K*	Ca*	Mg*	S*	Zn*	Cu*	Mn*	Fe*	Na*
pH	1	0.5003 0.0005	0.2961 0.0483	0.2878 0.0553	0.1112 0.4670	0.2501 0.0975	0.4712 0.0011	0.3029 0.0431	0.4248 0.0036	-0.1280 0.4021	0.3535 0.0172	0.4054 0.0057
N		1	0.7868 <.0001	0.7124 <.0001	0.6201 <.0001	0.6087 <.0001	0.7074 <.0001	0.6561 <.0001	0.7106 <.0001	0.3999 0.0065	0.3820 0.0096	0.6271 <.0001
P			1	0.8724 <.0001	0.7479 <.0001	0.7554 <.0001	0.7760 <.0001	0.7798 <.0001	0.8368 <.0001	0.6658 <.0001	0.6313 <.0001	0.6333 <.0001
K				1	0.7415 <.0001	0.9009 <.0001	0.8228 <.0001	0.7949 <.0001	0.8064 <.0001	0.6351 <.0001	0.5879 <.0001	0.6982 <.0001
Ca					1	0.8232 <.0001	0.6334 <.0001	0.8565 <.0001	0.7679 <.0001	0.7235 <.0001	0.5229 0.0002	0.6701 <.0001
Mg						1	0.7708 <.0001	0.8880 <.0001	0.7637 <.0001	0.7082 <.0001	0.5449 0.0001	0.7439 <.0001
S							1	0.7000 <.0001	0.6936 <.0001	0.4054 0.0057	0.4596 0.0015	0.6569 <.0001
Zn								1	0.8293 <.0001	0.7802 <.0001	0.6640 <.0001	0.7198 <.0001
Cu									1	0.5807 <.0001	0.7120 <.0001	0.7133 <.0001
Mn										1	0.6083 <.0001	0.5003 0.0005
Fe											1	0.4488 0.0020
Na												1

*Highlighted results indicate significant correlations at the 0.05 level.

Table 3. Spearman correlation coefficients for Piedmont loblolly pine xylem nutrient concentrations for the pre-burn period, Clemson Experimental Forest, SC, 2011.

	N	P*	K*	Ca*	Mg*	S*	Zn*	Cu*	Mn*	Fe	Na*
N	1	0.7643	0.6179	0.6929	0.5571	0.6321	0.7107	0.7107	0.2929	0.3071	0.6714
		0.0009	0.0141	0.0042	0.0310	0.0115	0.0030	0.0030	0.2895	0.2655	0.0061
P		1	0.7250	0.8286	0.6071	0.6000	0.7643	0.6393	0.5357	0.2429	0.5929
			0.0022	0.0001	0.0164	0.0181	0.0009	0.0103	0.0396	0.3831	0.0198
K			1	0.6607	0.9536	0.8964	0.7143	0.7393	0.0016	0.1250	0.8393
				0.0073	<.0001	<.0001	0.0028	0.0016	0.2318	0.6571	<.0001
Ca				1	0.6571	0.6214	0.6750	0.5893	0.5536	-0.0357	0.6250
					0.0078	0.0134	0.0058	0.0208	0.0323	0.8994	0.0127
Mg					1	0.8821	0.6857	0.7107	0.4250	0.0857	0.8500
						<.0001	0.0048	0.0030	0.1143	0.7613	<.0001
S						1	0.7464	0.6964	0.3107	0.2786	0.8429
							0.0014	0.0039	0.2597	0.3147	<.0001
Zn							1	0.7929	0.4321	0.3393	0.7536
								0.0004	0.1077	0.2160	0.0012
Cu								1	0.1679	0.3393	0.8714
									0.5499	0.2160	<.0001
Mn									1	0.2464	0.3464
										0.3760	0.2059
Fe										1	0.1679
											0.5499
Na											1

*Highlighted results indicate significant correlations at the 0.05 level.

Table 4. Spearman correlation coefficients for Piedmont loblolly pine xylem nutrient concentrations for the fire period, Clemson Experimental Forest, SC, 2011.

	N	P	K*	Ca*	Mg*	S*	Zn*	Cu*	Mn*	Fe*	Na*
N	1	0.4964	0.5429	0.3071	0.2071	0.6571	0.3607	0.2607	0.1679	0.3036	0.3071
		0.0598	0.0365	0.2655	0.4588	0.0078	0.1866	0.3480	0.5499	0.2714	0.2655
P		1	0.5714	0.5429	0.4964	0.5429	0.7321	0.5893	0.5286	0.7500	0.3036
			0.0261	0.0365	0.0598	0.0365	0.0019	0.0208	0.0428	0.0013	0.2714
K			1	0.1464	0.5893	0.5679	0.6214	0.4893	0.3143	0.6393	0.4214
				0.6025	0.0208	0.0272	0.0134	0.0642	0.2539	0.0103	0.1177
Ca				1	0.3286	0.2036	0.5750	0.6893	0.7036	0.6071	0.1821
					0.2318	0.4668	0.0249	0.0045	0.0034	0.0164	0.5159
Mg					1	0.2357	0.8500	0.6357	0.5000	0.6857	0.4893
						0.3977	<.0001	0.0109	0.0577	0.0048	0.0642
S						1	0.4857	0.4679	-0.1214	0.4107	0.4250
							0.0664	0.0786	0.6664	0.1283	0.1143
Zn							1	0.8179	0.5964	0.8714	0.5143
								0.0002	0.0189	<.0001	0.0498
Cu								1	0.6071	0.7893	0.3857
									0.0164	0.0005	0.1556
Mn									1	0.6393	0.0786
										0.0103	0.7808
Fe										1	0.4143
											0.1247
Na											1

*Highlighted results indicate significant correlations at the 0.05 level.

Table 5. Spearman correlation coefficients for Piedmont loblolly pine xylem nutrient concentration for the post-burn period, Clemson Experimental Forest, SC, 2011.

	N*	P*	K*	Ca*	Mg*	S*	Zn*	Cu*	Mn*	Fe*	Na*
N	1	0.7679 0.0008	0.7464 0.0014	0.6893 0.0045	0.7143 0.0028	0.6464 0.0092	0.6714 0.0061	0.7096 0.0030	0.3929 0.1475	0.4750 0.0736	0.7643 0.0009
P		1	0.8679 <.0001	0.8714 <.0001	0.8179 0.0002	0.7179 0.0026	0.6714 0.0061	0.8865 <.0001	0.5536 0.0323	0.7036 0.0034	0.7286 0.0021
K			1	0.7321 0.0019	0.9107 <.0001	0.7893 0.0005	0.6071 0.0164	0.8454 <.0001	0.4643 0.0813	0.6393 0.0103	0.6357 0.0109
Ca				1	0.7429 0.0015	0.6536 0.0082	0.8000 0.0003	0.7685 0.0008	0.6214 0.0134	0.6893 0.0045	0.6429 0.0097
Mg					1	0.8250 0.0002	0.7679 0.0008	0.7954 0.0004	0.5536 0.0323	0.6500 0.0087	0.6607 0.0073
S						1	0.5929 0.0198	0.6667 0.0066	0.2679 0.3344	0.5464 0.0351	0.6107 0.0156
Zn							1	0.7382 0.0017	0.7571 0.0011	0.7643 0.0009	0.5500 0.0337
Cu								1	0.6971 0.0039	0.8794 <.0001	0.6291 0.0120
Mn									1	0.6464 0.0092	0.3893 0.1515
Fe										1	0.4679 0.0786
Na											1

*Highlighted results indicate significant correlations at the 0.05 level.

Site Modeling Analysis of Variance and Characteristics

The results suggest differences between sites and soil were negligible, but vegetation composition was influential to concentrations of some of the nutrients (Tables 6 and 7). Although not a true blocking factor, the site replicate factors (sites) for soil and duff nutrients were not significant, regardless of model. None of the soil nutrients differed between sites (4 burned and 1 unburned sites). Based on the nutrient sufficiency levels provided in the soil analysis, phosphorus was the most limiting nutrient measured for all sites, with concentrations ranging from 4.4 to 5.5 lbs/ac. However, all nutrient concentrations tended to be lower than the Clemson Agricultural Services Lab recommended concentrations for loblolly pine.

Of the eight duff nutrients analyzed, only calcium differed between treatments. The concentration of calcium in the control treatment (7419.3 ppm, ±1130.3 SE) was more than double that of the fire treatment (3118.6 ppm, ±565.2 SE) for duff, $F(3, 10) = 11.58$, $P = 0.0424$). Because none of the other nutrients differed between treatments, the decrease in calcium concentration in the fire treatment is not likely the result of burning. Calcium content in Piedmont forests has been reported to be very sensitive to differences in vegetation composition (Metz, 1952). Sites with a hardwood component can have two to six times the calcium content of pure pine sites, and the control site was observed to have the most significant hardwood component of any of the sites analyzed (Metz, 1952).

Table 6. Mixed models analysis of variance results for soil nutrient concentrations and standard errors in Piedmont loblolly pine stands by burning treatment, Clemson Experimental Forest, SC, 2011.

Effect	*df*	*F*	*P*	Fire (lbs/ac)*	Control (lbs/ac)*
P	3, 1	0.96	0.3998	4.480 ± 0.4917a	5.528 ± 0.9513a
K	3, 1	3.10	0.1763	58.982 ± 5.563a	73.110 ± 11.126a
Ca	3, 1	6.48	0.0843	248.12 ± 36.821a	501.92 ± 104.74a
Mg	3, 1	2.26	0.2301	57.332 ± 5.844a	78.646 ± 13.690a
Zn	3, 1	0.86	0.4212	8.503 ± 7.082a	3.246 ± 14.164a
Cu	3, 1	0.02	0.8964	5.446 ± 4.691a	1.017 ± 9.382a
Mn	3, 1	0.01	0.9380	63.258 ± 13.479a	65.803 ± 26.957a
Na	3, 1	0.01	0.9192	7.917 ± 1.691a	8.333 ± 3.382a

* Results of post-ANOVA Tukey's significant difference test. Same letter indicates no significant difference between columns detected between treatments at the 0.05 level.

Table 7. Mixed models analysis of variance results for duff nutrient concentrations and standard errors in Piedmont loblolly pine stands by burning treatment, Clemson Experimental Forest, SC, 2011.

Effect	*df*	*F*	*P*	Fire (ppm)*	Control (ppm)*
P	3, 1	9.76	0.0523	469.1 ± 016.2a	582.3 ± 032.4a
K	3, 1	0.14	0.7345	798.2 ± 121.4a	697.2 ± 242.7a
Ca	3, 1	11.58	0.0424	3118.6 ± 565.2a	7419.3 ± 1130.3b
Mg	3, 1	0.36	0.5907	709.8 ± 150.6a	912.0 ± 301.3a
Zn	3, 1	0.02	0.8856	8.606 ± 2.070a	9.346 ± 4.315a
Cu	3, 1	4.15	0.1345	534.08 ± 102.51a	1000.97 ± 205.02a
Mn	3, 1	0.0	0.9585	8045.36 ± 1768.42a	8247.03 ± 3211.92a
Na	3, 1	0.01	0.9412	38.371 ± 5.767a	29.403 ± 11.533a

* Results of post-ANOVA Tukey's significant difference test. Same letter indicates no significant difference between columns detected between treatments at the 0.05 level.

Immobile Nutrients Analysis of Variance

Relationships tended to be similar across all standardized nutrient concentrations. Of the five immobile nutrients analyzed using ANOVA (Ca, Fe, Zn, Mn, and Cu), only Fe did not differ between periods (Table 8). None of the treatment or interaction factors were significant, which is primarily attributed to high variability resulting from multiple year resolution.

Calcium concentrations differed across all periods with concentrations increasing from the pre-burn (101.7 ppm ± 16.3 SE) to the fire period (118.0 ppm ± 18.9 SE), then decreasing in the post-burn (81.5 ppm ± 13.0 SE) period, $P < 0.001$, $F(36,2) = 26.24$ (Table 8). Similarly, Zn differed across periods with concentrations increasing from the pre-burn (159.4 ppm ± 32.1 SE) to the fire period (230.5 ppm ± 32.1 SE), then decreasing in the post-burn (108.6 ppm ± 32.1 SE) period, $P < 0.001$, $F(36,2) = 18.84$. Copper and manganese concentrations only differed between the fire period (0.432 ppm ± 0.118 SE and 20.116 ppm ± 4.297 SE, respectively) and the post-burn period (0.220 ppm ± 0.118 SE and 9.468 ppm ± 4.297 SE, respectively), but had a similar trend to the other immobile nutrients with the fire period having the greatest concentration of any of the periods. Iron concentrations did not statistically differ across periods.

Although not significant, Ca, Zn, and Cu had slightly higher concentrations in the control treatment than the burn treatments, while Mn and Fe had the inverse relationship (Table 8). In addition, many nutrients had higher concentrations in the interactions for fire treatment, fire period than the control treatment for the same period. The pre- and post-burn nutrient concentrations for both treatments tended to be similar. However, the fire period tended to have higher concentrations than the pre-burn and post-burn periods regardless of treatment, with the exception of Fe. Iron tended to have a three-fold increase in the fire treatment, fire period while all of the other observations had similar nutrient concentrations.

Table 8. Mixed models analysis of variance results for Piedmont loblolly pine xylem immobile nutrient concentrations and standard errors by treatment, period, and interaction with covariates, Clemson Experimental Forest, SC, 2011.

	Ca (ppm)*	Zn (ppm)*	Cu (ppm)*	Mn (ppm)*	Fe (ppm)
Treatment	$F(3, 1) = 1.09$	$F(3, 1) = 0.25$	$F(3, 1) = .69$	$F(3, 1) = 0.03$	$F(3, 1) = 0.28$
Fire	85.9 ± 12.3	1.512 ± 0.268	0.298 ± 0.085	15.112 ± 3.502	4.129 ± 1.838
Control	117.0 ± 28.8	1.811 ± 0.536	0.349 ± 0.169	13.693 ± 7.005	2.247 ± 2.711
Period	$F(36, 2) = 26.24$	$F(36, 2) = 18.84$	$F(36, 2) = 4.61$	$F(36, 2) = 6.12$	$F(36, 2) = 2.04$
Pre-burn	101.7 ± 16.3a	1.594 ± 0.321a	0.318 ± 0.118ab	13.6254 ± 4.297ab	2.950 ± 1.803
Fire	118.0 ± 18.9b	2.305 ± 0.321b	0.432 ± 0.118a	20.116 ± 4.297a	4.128 ± 2.132
Post-burn	81.5 ± 13.0c	1.086 ± 0.321c	0.220 ± 0.118b	9.468 ± 4.297b	2.393 ± 1.624
Interaction	$F(36, 2) = 2.77$	$F(36, 1) = 3.00$	$F(36, 2) = 2.84$	$F(36, 2) = 0.82$	$F(36, 2) = 3.29$
F-Pre	79.1 ± 12.7	1.213 ± 0.287	0.189 ± 0.106	13.229 ± 3.844	3.568 ± 1.773
F-Fire	136.5 ± 16.7	2.410 ± 0.287	0.524 ± 0.106	23.097 ± 3.844	7.005 ± 2.485
F-Post	52.2 ± 10.3	0.914 ± 0.287	0.180 ± 0.106	9.013 ± 3.844	2.434 ± 1.465
C-Pre	131.2 ± 32.7	1.975 ± 0.574	0.447 ± 0.211	14.022 ± 7.687	2.390 ± 2.903
C-Fire	142.2 ± 34.1	2.199 ± 0.574	0.340 ± 0.211	17.135 ± 7.687	2.007 ± 2.660
C-Post	82.4 ± 25.9	1.259 ± 0.574	0.260 ± 0.211	9.923 ± 7.687	2.353 ± 2.880
Covariate					
pH	N/A	N/A	N/A	N/A	N/A

* Results of post-ANOVA Tukey's significant difference test. Same letter indicates no significant difference within columns detected between treatments at the 0.05 level.

Mobile Nutrients Analysis of Variance

Relationships among the mobile nutrients were similar to that of the immobile nutrients (Table 9). Concentrations of N, P, K, Mg and S differed by periods. Nitrogen was the only nutrient to differ by treatments with the concentrations in the control (0.042 ppm ± 0.006 SE) over 2.5 times greater than the fire treatments (0.015 ppm ± 0.003 SE). None of the interactions were significant. Sulfur and sodium were the only mobile nutrients to have a significant covariate factors.

Nitrogen concentrations pre-burn (0.022 ppm ± 0.004 SE) and post-burn (0.026 ppm ± 0.004 SE) differed from the fire period (0.037 ppm ± 0.004 SE). Fire period concentrations of K (153.0 ppm ± 25.3 SE) and Mg (47.4 ppm ± 5.0 SE) were also significantly greater than pre-burn (99.8 ppm ± 25.3 SE and 31.8 ppm ± 5.0 SE, respectively) and post-burn (101.5 ppm ± 25.3 SE and 25.2 ppm ± 5.0 SE, respectively) concentrations. Phosphorus concentrations had a similar trend with the fire period (13.9 ppm ± 3.1 SE) having greater concentrations than pre-burn (7.6 ppm ± 2.3 SE) and post-burn (10.5 ppm ± 2.7 SE) concentrations but only pre-burn concentrations differed from the fire period. Concentrations of S in the fire period (19.7 ppm ± 2.5 SE) differed from the pre-burn (10.0 ppm ± 2.5 SE) and post-burn (13.2 ppm ± 2.5 SE) periods, $P = 0.002$, $F(35.2,2) = 11.03$. Sodium concentrations did not significantly differ, but had similar trends in the burned and unburned sites as the immobile nutrients. The pH covariate was significant for S and Na suggesting soil pH has more significant influences on S and Na concentrations than burning.

Table 9. Mixed models analysis of variance results for Piedmont loblolly pine xylem mobile nutrient concentrations by treatment, period, and interaction with covariates, Clemson Experimental Forest, SC, 2011.

Effect	N (ppm)*	P (ppm)*	K (ppm)*	Mg (ppm)*	S (ppm)*	Na (ppm)
Treatment	$F(3, 1) = 13.78$	$F(3, 1) = 0.80$	$F(3, 1) = 0.22$	$F(3, 1) = 0.32$	$F(3.22, 1) = 0.11$	$F(3.09, 1) = 0.47$
Fire	0.015 ± 0.003a	8.4 ± 2.1	106.7 ± 21.9	32.3 ± 4.0	13.6 ± 1.9	2.857 ± 0.384
Control	0.042 ± 0.006b	12.9 ± 5.1	129.5 ± 43.8	37.3 ± 8.0	15.1 ± 3.9	3.549 ± 0.974
Period	$F(36, 2) = 11.49$	$F(36, 2) = 9.87$	$F(36, 2) = 14.56$	$F(36, 2) = 15.94$	$F(35.2, 2) = 11.03$	$F(35.3, 2) = 1.67$
Pre-burn	0.022 ± 0.004a	7.6 ± 2.3a	99.8 ± 25.3a	31.8 ± 5.0a	10.0 ± 2.5a	3.089 ± 0.676
Fire	0.037 ± 0.004b	13.9 ± 3.1b	153.0 ± 25.3b	47.4 ± 5.0b	19.7 ± 2.5b	4.134 ± 0.873
Post-burn	0.026 ± 0.004a	10.5 ± 2.7ab	101.5 ± 25.3a	25.2 ± 5.0a	13.2 ± 2.5a	2.513 ± 0.568
Interaction	$F(36, 2) = 0.96$	$F(36, 2) = 2.89$	$F(36, 2) = 2.18$	$F(36, 2) = 1.04$	$F(35.2) = 0.15$	$F(35.3, 2) = 1.21$
F-Pre	0.011 ± 0.004	5.7 ± 1.8	84.1 ± 22.7	28.0 ± 4.5	9.3 ± 2.2	2.538 ± 0.508
F-Fire	0.021 ± 0.004	13.3 ± 2.7	154.9 ± 22.7	48.2 ± 4.5	19.6 ± 2.2	4.702 ± 0.871
F-Post	0.013 ± 0.004	7.0 ± 1.9	81.2 ± 22.7	20.6 ± 4.5	11.9 ± 2.2	1.895 ± 0.401
C-Pre	0.034 ± 0.007	9.8 ± 4.6	115.6 ± 45.3	35.6 ± 9.0	10.7 ± 4.5	3.741 ± 1.454
C-Fire	0.052 ± 0.007	14.5 ± 5.6	151.2 ± 45.3	46.6 ± 9.0	19.9 ± 4.5	3.628 ± 1.416
C-Post	0.040 ± 0.007	14.8 ± 5.7	121.8 ± 45.3	29.8 ± 9.0	14.6 ± 4.5	3.291± 1.230
Covariate					$F(36.8, 1) = 8.58$	$F(17.4, 1) = 4.93$
pH	N/A	N/A	N/A	N/A	12.3 ± 4.2	0.987 ± 0.445

*Results of post-ANOVA Tukey's significant difference test. Same letter indicates no significant difference within columns detected between treatments at the 0.05 level.

Multivariate Analysis of Variance

MANOVA modeling resulted in fewer significant nutrient variables than the ANOVA modeling. The significant variables were variables with the strongest univariate relationships. MANOVA tends to report less significance between variables than ANOVA because MANOVA removes the correlation between each of the variables and is more conservative regarding family-wise errors. Because the Bonferroni correction was used, the individual alphas differed by model.

Analyses of all nutrients (N, P, K, Ca, Mg, S, Zn, Cu, Mn, Fe, and Na) suggested N was the only concentration to differ by treatments (Table 10). Nitrogen concentrations in the control treatment (0.0417 ppm ± 0.010 SE) were over two and a half times the concentrations in the fire treatment (0.015 ppm ± 0.010 SE). Covariates were not significant.

Each period differed for Ca, Zn, S, N, and Mg (Table 10). Calcium increased from pre-burn (90.8 ppm ± 30.0 SE) to the fire (140.5 ppm ± 39.9 SE) period then decreased to less than pre-burn concentration by the post-burn (60.2 ppm ± 24.3 SE) period, $P < 0.001$, $F(68, 2) = 14.199$. Sulfur also increased from pre-burn (9.6 ppm ± 4.3 SE) to the fire (19.6 ppm ± 7.6 SE) period, then decreased to greater than pre-burn levels by the post-burn (12.4 ppm ± 6.8 SE) period, $P = 0.0060$, $F(68, 2) = 5.895$. Similar to Ca, zinc increased from pre-burn (1.365 ppm ± 0.543 SE) to the fire (2.368 ppm ± 0.812 SE) period, then decreased to less than pre-burn levels by the post-burn (0.983 ppm ± 0.466 SE) period, $P < 0.0001$, $F(68, 2) = 9.592$. Both N and Mg differed between all periods and had a similar trend. Nitrogen concentrations increased from pre-burn (0.016 ppm ± 0.011 SE) to the fire period (0.028 ppm ± 0.017 SE), then decreased by the post-burn period (0.018 ppm ± 0.013 SE). Concentrations of magnesium also increased from the pre-burn (29.5 ppm ± 4.9 SE) to the fire period (47.9 ppm ± 12.6 SE) but decreased to lower concentrations than the pre-burn level by the post-burn period (22.4 ppm ± 9.3 SE).

Table 10. Multivariate analysis of variance results using repeated measures generalized linear models analysis for Piedmont loblolly pine xylem nutrient concentrations and standard deviations by treatment, period, and interaction with covariates, Clemson Experimental Forest, SC, 2011.

Dependent Variable	Effect	df	F	Sig.	Period	Fire Mean	Fire SD	Control Mean	Control SD	Period Total Mean	Period Total SD
N	Treatment*	1	54.838	0.000	Pre	0.011	0.001	0.036	0.004	0.015	0.011
	Period	2	7.386	0.002	Fire	0.021	0.012	0.052	0.006	0.028	0.006
	pH	1	0.386	0.538	Post	0.013	0.007	0.040	0.007	0.018	0.013
					Total*	0.015	0.010	0.042	0.010	0.021	0.014
P	Treatment	1	2.835	0.100	Pre	5.9	2.1	10.0	3.6	6.7	2.8
	Period	2	4.088	0.025	Fire	14.3	7.8	14.6	1.6	14.4	6.9
	pH	1	0.060	0.808	Post	7.4	3.7	15.1	4.8	8.9	4.9
					Total	9.2	6.2	13.2	3.9	10.0	6.0
K	Treatment	1	1.262	0.268	Pre	84.0	38.1	115.6	23.4	90.4	37.3
	Period	2	4.536	0.017	Fire	154.9	50.0	151.1	11.8	154.1	44.6
	pH	1	0.009	0.924	Post	81.2	50.0	121.8	24.7	89.3	48.3
					Total	106.7	56.8	130.0	24.4	111.2	52.5
Mg	Treatment	1	0.537	0.468	Pre	28.0	10.8	35.6	4.9	29.5	10.2
	Period	2	10.534	0.000	Fire	48.2	13.6	46.6	9.2	47.9	12.6
	pH	1	0.356	0.554	Post	20.6	9.4	29.8	4.9	22.4	9.3
					Total	32.3	16.2	37.3	9.4	33.3	15.1
Na	Treatment	1	0.123	0.727	Pre	2.773	1.622	4.703	1.011	3.159	1.688
	Period	2	0.614	0.546	Fire	6.799	9.472	4.525	0.677	6.344	8.452
	pH	1	0.024	0.877	Post	2.036	1.140	4.228	1.341	2.474	1.450
					Total	3.870	5.826	4.486	0.929	3.993	5.217
S	Treatment	1	2.004	0.165	Pre	8.7	3.5	13.1	6.0	9.6	4.3
	Period	2	5.758	0.007	Fire	19.0	7.5	22.3	8.9	19.6	7.6
	pH	1	0.093	0.762	Post	11.3	6.9	16.9	4.2	12.4	6.8
					Total	13.0	7.5	17.4	7.0	13.9	7.5

*Results of post-MANOVA Tukey's significant difference test. *Indicates difference detected at the 0.05 level.

Table 10 (Continued). Multivariate analysis of variance results using repeated measures generalized linear models analysis for Piedmont loblolly pine xylem nutrient concentrations and stand deviations by treatment, period, and interaction with covariates, Clemson Experimental Forest, SC, 2011.

Dependent Variable						Fire		Control		Period Total	
	Effect	df	F	Sig.	Period	Mean	SD	Mean	SD	Mean	SD
Ca	Treatment	1	4.543	0.040	Pre	80.4	19.9	132.4	29.3	90.8	30.0
	Period	2	13.840	0.000	Fire	140.0	44.5	142.4	14.5	140.5	39.9
	pH	1	0.013	0.911	Post	54.5	23.0	82.9	15.8	60.2	24.3
					Total	91.6	47.2	119.2	33.0	97.2	45.8
Zn	Treatment	1	0.419	0.521	Pre	1.213	0.455	1.975	0.478	1.366	0.543
	Period	2	9.523	0.000	Fire	2.410	0.897	2.199	0.367	2.368	0.812
	pH	1	0.721	0.401	Post	0.914	0.489	1.259	0.254	0.983	0.466
					Total	1.512	0.907	1.811	0.537	1.572	0.849
Cu	Treatment	1	0.282	0.599	Pre	0.189	0.126	0.447	0.236	0.241	0.178
	Period	2	1.228	0.304	Fire	0.524	0.515	0.400	0.024	0.487	0.463
	pH	1	0.067	0.798	Post	0.180	0.110	0.260	0.052	0.196	0.105
					Total	0.298	0.344	0.349	0.146	0.308	0.314
Mn	Treatment	1	0.095	0.760	Pre	13.229	6.445	14.022	6.253	13.387	6.191
	Period	2	3.562	0.038	Fire	23.097	12.971	17.135	9.541	21.904	12.300
	pH	1	0.012	0.915	Post	9.012	4.780	9.923	8.086	9.195	5.238
					Total	15.113	10.438	13.693	7.661	14.829	9.882
Fe	Treatment	1	1.806	0.187	Pre	4.060	2.944	2.460	1.050	3.740	2.721
	Period	2	0.756	0.477	Fire	8.575	7.852	2.015	0.316	7.263	7.472
	pH	1	0.002	0.965	Post	3.009	2.904	2.501	1.490	2.908	2.644
					Total	5.215	5.545	2.325	0.954	4.637	5.098

*Results of post-MANOVA Tukey's significant difference test. *Indicates difference detected at the 0.05 level.

CHAPTER VII
DISCUSSION FOR FIRE SIGNATURE DETECTION USING ICP-MS IN LOBLOLLY PINE, CLEMSON EXPERIMENTAL FOREST, SC, USA

Nutrient Modeling

Results of nutrient modeling were congruent across all analyses. The results suggest the nutrient concentration relationships vary through time. Using univariate and multivariate analyses provided more robust results, and reinforced the accuracy of the results. Analyzing differences in the means, as opposed to the relationships between the variables, is both a statistically more powerful test and provides better clues to identifying a potential fire signature. However, the change in the trend of the relationships combined with the limited temporal resolution restricted the possible analyses and ability to adequately develop a model to identify a fire signature. Finer temporal resolution would have allowed for time series modeling, cluster and factor analyses, or regression trees.

Nutrient Dynamics

Trees are not passive recorders of environmental change, and tree physiological changes occur both as trees mature and as a result of changes in environmental conditions (Smith and Shortle, 1996). Dendrochemical research is limited, and preferential transport and exclusion of nutrients are not fully understood for all tree species. The ANOVA and MANOVA results in this study suggest nutrients in Piedmont loblolly pine primarily differed by periods, but some of the non-significant interactions indicate nutrient concentrations may differ if replicated with finer resolution and increased sample size of the unburned stands.

Mobile nutrients are typically the most limited nutrients in loblolly pine ecosystems (Adams and Allen, 1985). Concentrations of mobile nutrients primarily differ through time because mobile nutrients are transported from areas of excess to areas of deficiency as required for growth and maintenance. The results of this study indicate the fire period had the greatest concentrations of all mobile nutrients regardless of treatment, except for nitrogen.

Because there was only one control treatment stand, the potential differences between interactions may be masked by low statistical power resulting from larger variation in the unburned stand. Although mean treatment differences were not significant, they tended to be greater in control treatment than the fire treatments. These greater concentrations are primarily attributed to decreased nutrient pools resulting from fire, but may be a result of physiological changes, natural variation, sampling error, or other environmental changes.

Overall, mobile nutrient concentrations were well below the loblolly pine foliar nutrient guidelines for both the fire and unburned treatments (Jokela, 2004). Nutrient concentrations in xylem tend to be lower than that of foliar concentrations. Soil sampling suggested mobile nutrients, particularly P, were limiting in the stands. Sodium was influenced by soil pH, but soil, duff, and xylem concentrations were well below concentrations that would affect cation exchange capacity and within-tree nutrient availability.

Although interactions did not differ statistically, the low statistical power of the analyses resulting from small sample size presents the potential for speculation of mean differences. The interactions in the mobile nutrients tended to have the greatest concentrations of nutrients in the fire period for both fire and control treatments, but the control treatments had greater overall mean concentrations than the pre-burn and post-burn periods than the fire treatments. The mean difference between period differences was greater for the fire treatments, indicating a greater than expected increase in the fire period. Because the fire periods and tree ages differed by stand, this greater than expected increase in nutrients in the fire treatment, fire period is attributed to the short-term, fire fertilization effect combined with physiological changes resulting from fire-induced injury and not some other environmental change.

Fire appears to have more influence on select immobile nutrient concentrations than on mobile concentrations. Mean treatment differences of fire treatment Ca, Zn, and Cu had slightly lower concentrations than the control treatment, but the reverse relationship was present for Mn and Fe. Similar to the mobile nutrient concentrations, differences between periods were greatest in the fire period regardless of treatment. Similar to the mobile nutrient concentrations, immobile concentrations in the xylem and soils were also below the loblolly pine foliar nutrient guidelines for both the fire and control treatments suggesting immobile nutrients are also limiting in the stands sampled (Jokela, 2004).

The interaction relationships for the immobile nutrients (S, Zn, Cu, and Fe) differed from that of the mobile nutrients, but differences in S and Zn concentrations do not appear to be influenced by fire. Sulfur had slight increases in the fire period concentrations for both fire and control treatments, but concentrations between treatments were similar across all treatments and periods suggesting differences in periods were not a result of fire. Sulfur appears to be more sensitive to anthropogenic activities that alter atmospheric deposition (Kern et al., 2009). Concentrations of Zn had the same relationship between treatments and only differed through time, also suggesting differences in periods were also not a result of fire.

The Ca interaction had greater overall concentrations in the control treatment, but a more significant increase than expected in the fire period concentrations. Copper concentrations were greater in the control treatment and slightly decreased in concentration through time, while fire treatment, fire period concentrations were double that of the other periods. Interaction concentrations of Mn were similar for the pre-burn

and post-burn periods regardless of treatment, but the fire treatment, fire period was greater than the control treatment fire period. Iron had the most apparent interaction concentration differences in relationships. The control treatment was similar across all periods, but the fire treatment, fire period was more than double the control treatment concentrations and the other fire treatment periods. Similar to the mobile nutrient concentrations, the fire periods differed by stand and this greater than expected increase in fire treatment, fire period nutrient concentrations is attributed to the fire treatment.

Appropriateness of Technique and Resolution

The ICP-MS technique used had a sufficient detection limit to identify mean differences in nutrient concentration as a result of fire in Piedmont loblolly pine xylem. However, the methodology used was less than ideal for identifying a fire signature. The ICP-MS method required a minimum of two grams of plant material sample to accurately detect nutrient concentrations for standard foliar samples. The loblolly pine needle standard assumes samples have a significantly higher nutrient concentration than xylem, and as a result the detection resolution may not have been sensitive enough to minimize variability in xylem.

In addition to detection resolution issues, there was also variation in the concentrations resulting from inadequate sampling resolution. Because many of the rings were very small, to collect an adequate sample size, more rings were needed, and thus longer periods were sampled. For fire signature purposes, this variation in the number of rings used per period may have biased or diluted the signal to noise ratio. The ability to use annual resolution would provide more precise information regarding the magnitude of concentration differences and location and duration of changes in the nutrient concentrations.

Future studies should examine other techniques or extraction of the resinous fraction of the wood combined with a loblolly pine xylem standard at an annual resolution. More sensitive quantitative techniques, such as Liquid Chromatography-ICP-MS (LC-ICP-MS), offer better resolution with a smaller minimum samples size than ICP-MS. However many are more expensive and require extensive destructive sample preparation. Spectroscopic techniques would be ideal for resolving the sampling resolution problem, but are not quantitative. Calibration models would be required with the wet chemistry to provide quantitative concentrations, and would face similar sampling resolution difficulties as experienced in this study. Removal of the resinous fraction of wood prior to analyzing the nutrient composition could potentially increase the signal to noise ratio if the fire signature is contained within the cell walls. However, the resinous fraction of wood is composed of resin acids, fatty acids, terpene, alcohols, sterols, waxes, and resenes that require extensive destructive sample preparation prior to nutrient concentration analysis (Harris, 1952).

Temporal Consistency

The correlation analyses of the nutrient concentrations through time revealed inconsistencies in the nutrient concentration time relationship. Although most of the correlation analyses of the nutrient concentrations across all periods were significant, correlation analyses of the individual periods indicated the relationships and significance varied. Most of the nutrient concentration correlations showed a decreased correlation over the fire period. Because the individual fire years varied by stand and tree ages varied, this decrease is attributed to a change in the relationships between nutrients resulting from fire-induced altered nutrient pools and/or physiological responses to fire. Many macronutrients (such as N, Ca, K, Mg, S, and P) tend to accumulate with stand age, but the results did not find a proportional increase in nutrient concentrations (Jorgensen and Wells, 1986). Fire appears to temporarily shift the within-tree nutrient dynamics in these low intensity and severity fire systems.

Some nutrient correlations did not follow the trend of decreased correlations in the fire period. These deviations from the trend are primarily attributed to insufficient sampling resolution for those nutrients that are likely responsive to fire. However, some of the deviations could also be a result of differences in recovery rates of biogeochemical cycles, preferential transport and uptake of specific nutrients, physiological responses to fire, or non-fire related environmental changes. Because loblolly pine retains needles for two to three years, fire-induced increased nutrient availability potentially delays or extends growth responses similar to fertilization growth responses (Hynynen et al., 1998; Leggett and Kelting, 2006).

Fire Signature

ICP-MS analyses using intervals does not indicate a strong fire-nutrient signature for any single nutrient, except possibly iron. However, the results indicate the potential for a fire-nutrient signature using a combination of nutrient predictors. Similarly, dendrochemical studies of flood and volcano affected trees reported increases of immobile nutrients and rare earth elements in tree rings after the onset of a significant environmental disturbance (Pearson et al., 2009; St. George et al., 2006).

Unexpectedly, both mobile and immobile nutrients in xylem appear to have responded to fire, regardless of translocation and reallocation. A review of dendrochemical research shows that during the conversion of sapwood to heartwood, extensive translocation and reallocation occurs, generally resulting in disproportionally lower heartwood mineral concentrations (Meerts, 2002). Gymnosperms typically have an outwardly decreasing trend of nutrient concentrations. However, the results have greater than expected increases in concentrations of nutrients in the burned, fire period indicating the potential for modeling and predicting fire-induced nutrient changes in xylem.

Modeling and predicting fire-induced nutrient changes in loblolly pine xylem may vary by race and geography. Population differences exist by racial and geographic means in loblolly pine. Differences in growth, survival, rust resistance, cold hardiness, drought hardiness, disease resistance, and wood properties occur by geographic location and family (Dorman et al., 1973).

Assuming a suite of nutrients exists that consistently respond similarly to fire, the primary obstacle to overcome in modeling and predicting fire-induced nutrient changes in loblolly pine xylem will be overcoming differences in nutrient concentrations across different sites, such as differences in soil type and stand composition. Nitrogen or calcium concentrations may provide the best indicator of fire on a site, but will likely need to be adjusted to accommodate for these site differences.

As hypothesized, immobile nutrients (calcium, copper, and iron) currently appear to be responsive to fire. Although not statistically significant, this study results suggest Ca, Cu, and Fe may be the best variables for future investigation of identifying a fire signature because they are plant immobile and required for growth. Standardization of nutrient concentrations at a finer resolution will likely be necessary to identifying the suite of fire signature nutrients and account for site differences. More data are required to identify the suite of nutrients. At this time, it is uncertain if the potential exists to detect multiple fires, a fire signature in other species, or a fire signature in heartwood.

CHAPTER VIII
RESULTS FOR GROWTH-CLIMATE RESPONSE OF COASTAL PLAIN PLANTATION LOBLOLLY PINE, CLEMSON EXPERIMENTAL FOREST, SC, USA

Chronology Construction

A loblolly pine chronology with a total of 75 cores and cross-sections was used in the climate analysis with four tree ring series (here out 'series') removed because they did not correlate with the other series (Figure 3). The series removed tended to be complacent, suggesting that growth was either not limited or the individual trees were responding to factors other than regional climate, such as a localized disturbance (Fritts, 1976). The series removed were from two stands, Swine Center (series 2 and 7) and Outdoor Lab (series 5 and 10). The average interseries correlation was 0.505. The mean sensitivity was 0.260. First-order serial autocorrelation was 0.016.

The early to mid-1950s had below average ring widths. The early to mid-1920s may have had below average ring widths, but sample size was low for dates prior to 1940 ($n < 10$), so inferences on growth patterns should be made with discretion. The years 1970, 1981, and 1988 were those of slowest growth due to drought and these rings were often missing or micro-rings were present.

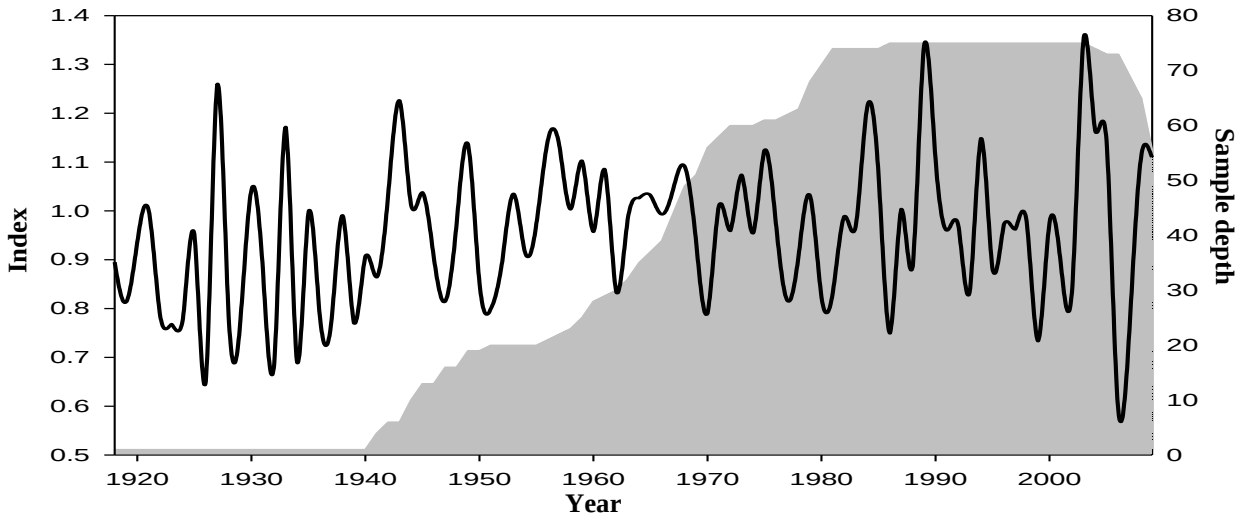

Figure 3. Residual chronology index spline (left Y-axis) for loblolly pine at the Clemson Experimental Forest for the period 1918 through 2008 (X-axis) Clemson Experimental Forest, SC, 2011. Sample depth area-fill by date is compared to the loblolly pine residual chronology (right Y-axis).

Correlation and Response Functions of Climate Variables

Correlation and response function analyses indicated late-summer, particularly July, had the most significant climate growth responses (Table 11). The master loblolly pine chronology exhibited correlations with temperature for previous September (r = –0.23, $P < 0.05$), current March (r = 0.19, $P < 0.05$), July (r = –0.37, $P < 0.05$), August (r = –0.30, $P < 0.05$), and November (r = 0.20, $P < 0.05$); however, only current July (r = –0.20, $P < 0.05$) was significant in the response function analysis (Figure 4). Precipitation was significantly correlated with growth in January (r = –0.23, $P < 0.05$), February (r = 0.27, $P < 0.05$), and July (r = 0.40, $P < 0.05$; Figure 4). Similar to temperature, July (r = 0.24, $P < 0.05$) was the only variable significant in the response function analysis**.**

Growth was positively correlated with PDSI for June (r = 0.22, $P < 0.05$), July (r = 0.32, $P < 0.05$), August (r = 0.24, $P < 0.05$), and September (r = 0.21, $P < 0.05$), with July (r = 0.20, $P < 0.05$) being the only significant variable for response function analysis (Figure 4). Similar to PDSI, growth was positively correlated with PHDI for July (r = 0.26, $P < 0.05$), August (r = 0.27, $P < 0.05$), September (r = 0.30, $P < 0.05$), October (r = 0.26, $P < 0.05$), and November (r = 0.22, $P < 0.05$), with August (r = 0.17, $P < 0.05$) being the only significant variable for response function analysis (Figure 4). The PHDI response was delayed because it accounts for the time lag associated with hydrologic processes. Combined, the correlation and response function results suggest this loblolly pine chronology has a strong late growing season moisture signal.

Table 11. Five most strongly correlated monthly variables, in ranked order, for loblolly pine growth on the Clemson Experimental Forest, Clemson, SC, 2011.

Variable	Correlation Function	Response Function
July Precipitation (+)	r = 0.40	r = 0.24
July Temperature (–)	r = –0.37	r = –0.20
July PDSI (+)	r = 0.32	r = 0.20
September PHDI (+)	r = 0.30	r = 0.17
August Temperature (+)	r = 0.30	Not significant

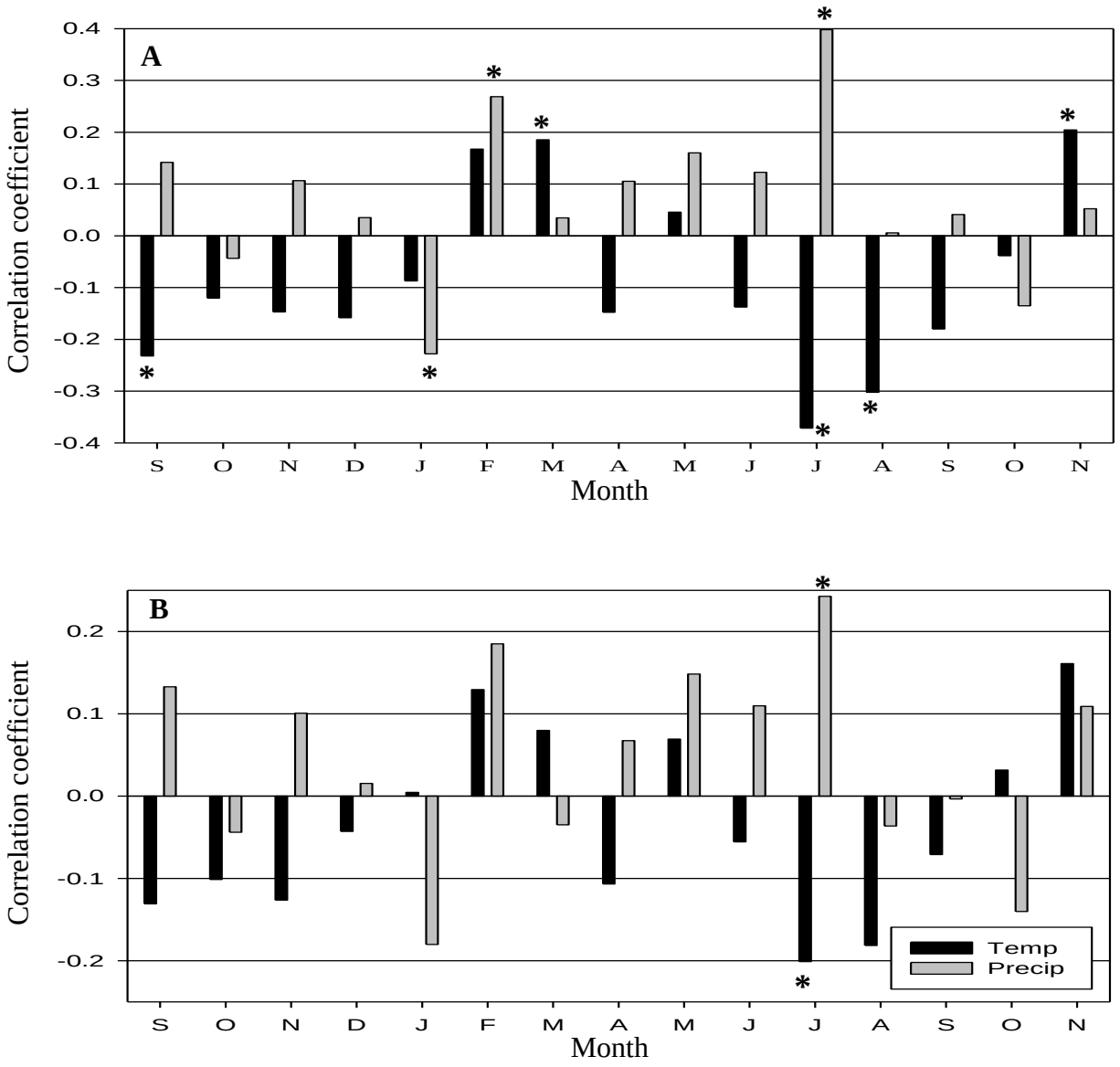

Figure 4. Correlation coefficients (Y-axis) of bootstrapped correlation (A) and response function (B) analyses between the Clemson Experimental Forest loblolly pine chronology and monthly mean temperature and total precipitation from the previous September to the current November (X-axis) from the period 1918 to 2008, Clemson Experimental Forest, SC, 2011. Asterisks (*) indicate significance at $P < 0.05$.

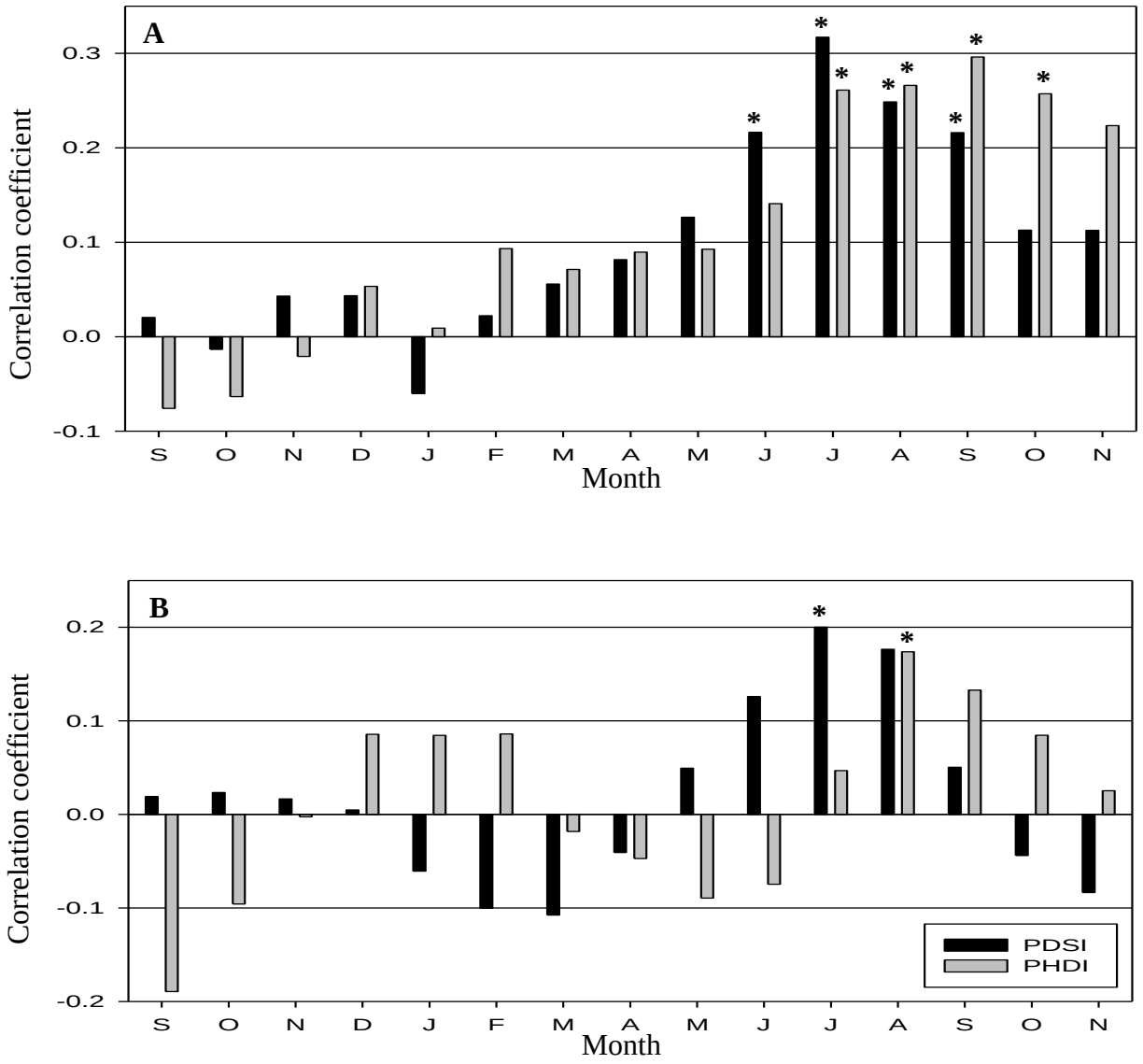

Figure 4. Correlation coefficients (Y-axis) of bootstrapped correlation (A) and response function (B) analyses between the Clemson Experimental Forest loblolly pine chronology and monthly mean PDSI and PHDI from the previous September to the current November (X-axis) from 1918 to 2008, Clemson Experimental Forest, SC, 2011. Asterisks (*) indicate significance at $P < 0.05$.

Moving Correlation Analysis of Climate Variables

Moving correlation analyses exhibited a temporal shift toward increased significance for all climate variables. Temperature signals were erratic for the periods prior to the 1948 to 1979 period, but have since been significantly correlated with growth (Figure 5). During the periods from 1918 to 1963, tree growth was moderately correlated with early growing season temperatures, but from the periods 1931 to 1977 the relationship degraded with some minor erroneous correlations present. Erroneous correlations result from inconsistent climate growth responses or Type I errors associated with multiple comparisons. Since the period 1945 to 1977, July temperatures have emerged as significant negative correlations with increasing strength of the relationship through time (Figure 6). Precipitation had a weak to nonexistent climate signal prior to the 1945 to 1977 period, but has since been significantly correlated with growth. Since the 1945 to 1977 period, many months, particularly July, have had significant positive correlations with growth and the intensity of the relationship has strengthened through time (Figures 7 and 8).

The relationships of drought indices had similar responses, and much like temperature and precipitation, were not significant prior to the period 1937 to 1969 (Figures 9 and 10). Both PDSI and PHDI had significant positive drought responses during the late growing season with increasing strength of the relationship through time, particularly in July for PDSI and August for PHDI (Figures 11 and 12). Weak significant negative correlations emerged from the period 1948 to 2000 for both PDSI and PHDI during the dormant season of the previous year. Similar to the correlation and response function analyses, moving correlation analyses identified a strong late growing season moisture sensitivity signal. The signal both been stable over time and the strength of the relationship has increased in strength with time.

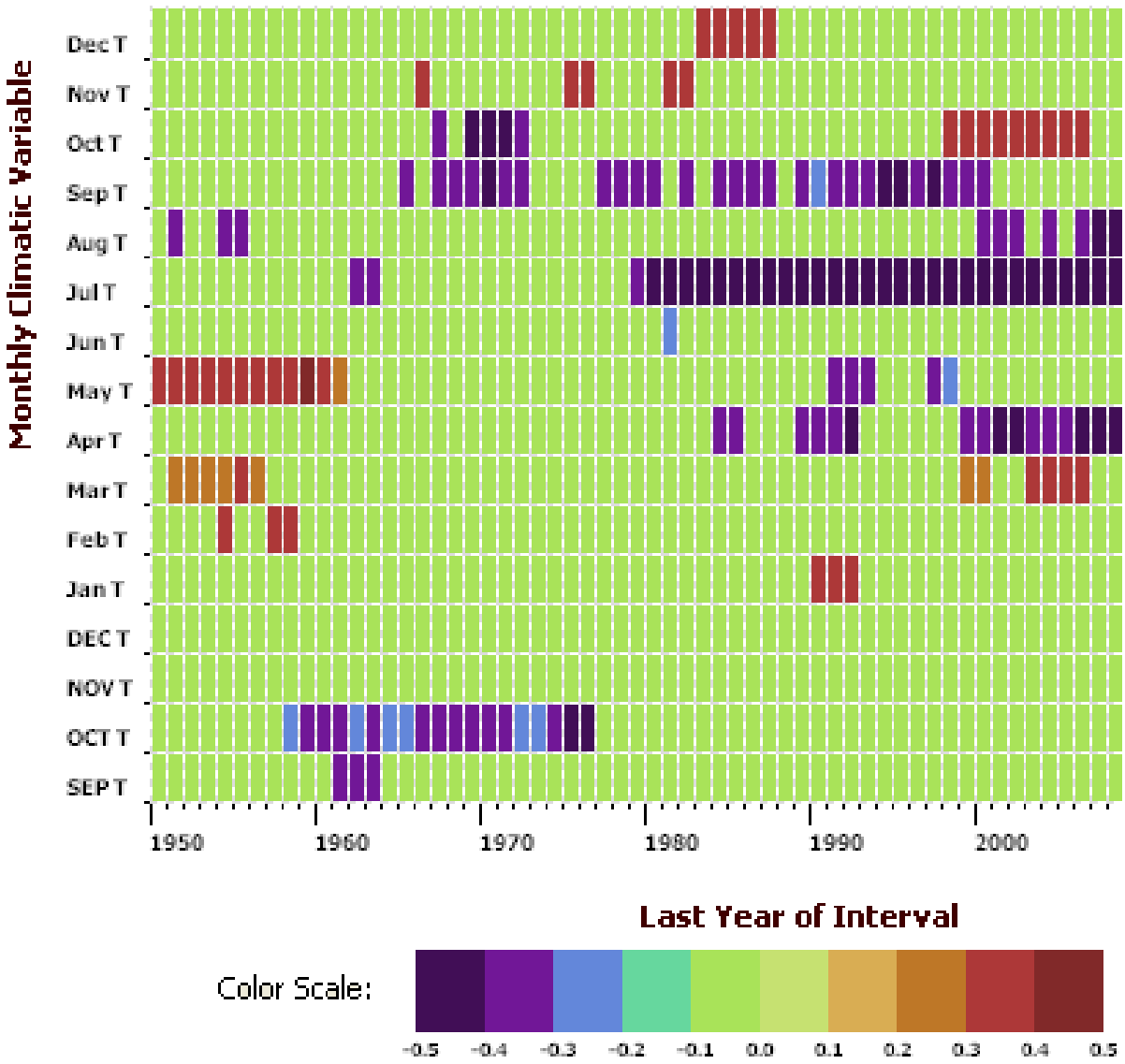

Figure 5. Moving correlation analysis between temperature and the Clemson Experimental Forest loblolly pine chronology, using 32-year moving intervals from the period 1918 to 2008, SC, 2011. Last year of moving intervals are listed on the x-axis. Grid squares marked 1950 represent the moving correlation coefficient by month for the period from 1918 to 1950. Non-significant correlation intervals are shaded green, while significant correlations are colored according to the strength and direction of the correlation coefficient.

Figure 6. Regressed temporal change in July temperature correlation coefficient significance (Y-axis) from 1918 to 2008 (X-axis) using 32-year moving intervals, Clemson Experimental Forest, SC, 2011. X-axis year marked 1950 represents the moving correlation coefficient by month for the period from 1918 to 1950.

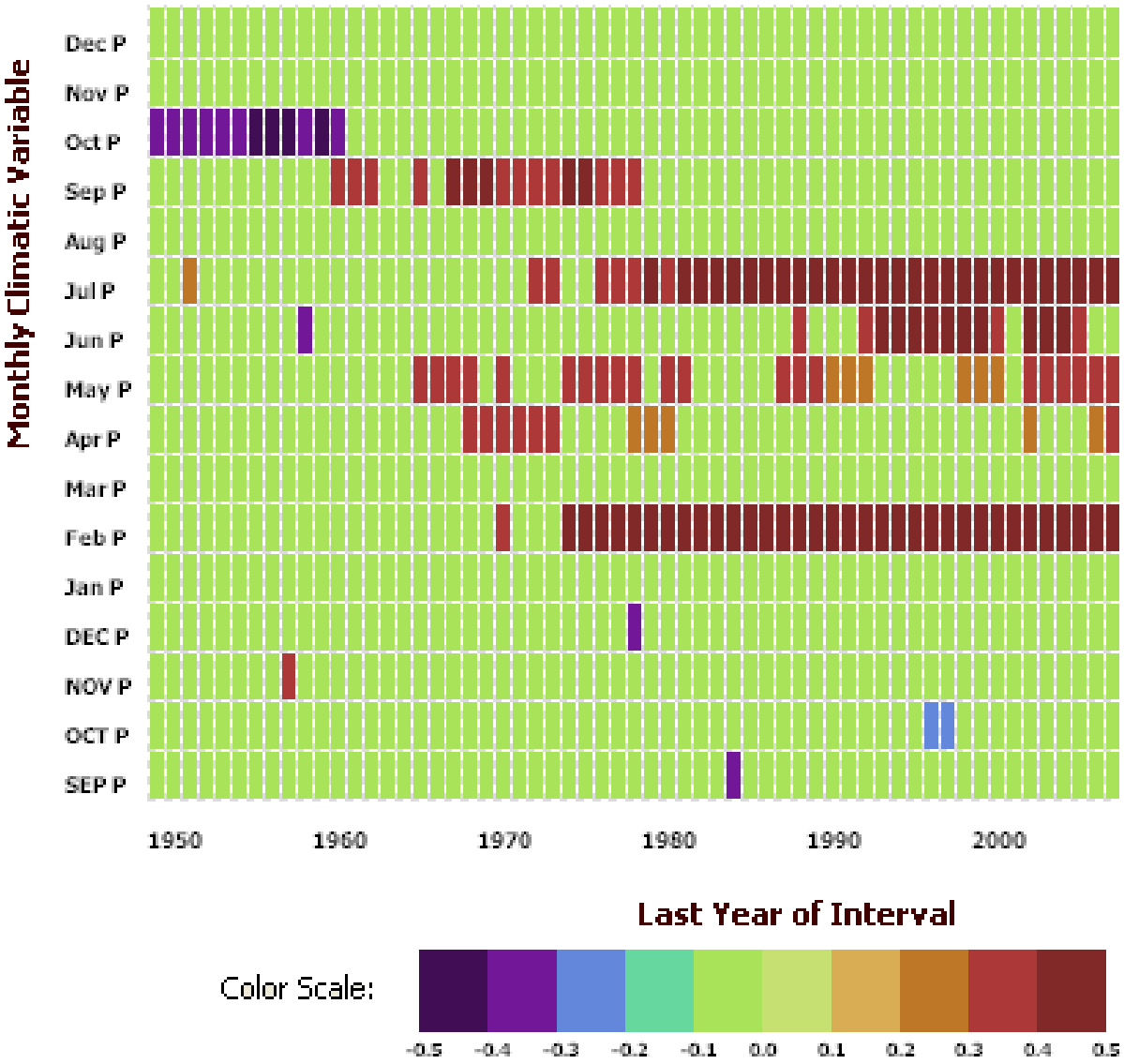

Figure 7. Moving correlation analysis between precipitation and the Clemson Experimental Forest loblolly pine chronology, using 32-year moving intervals from the period 1918 to 2008, SC, 2011. Last year of moving intervals are listed on the x-axis. Grid squares marked 1950 represent the moving correlation coefficient by month for the period from 1918 to 1950. Non-significant correlation intervals are shaded green, while significant correlations are colored according to the strength and direction of the correlation coefficient.

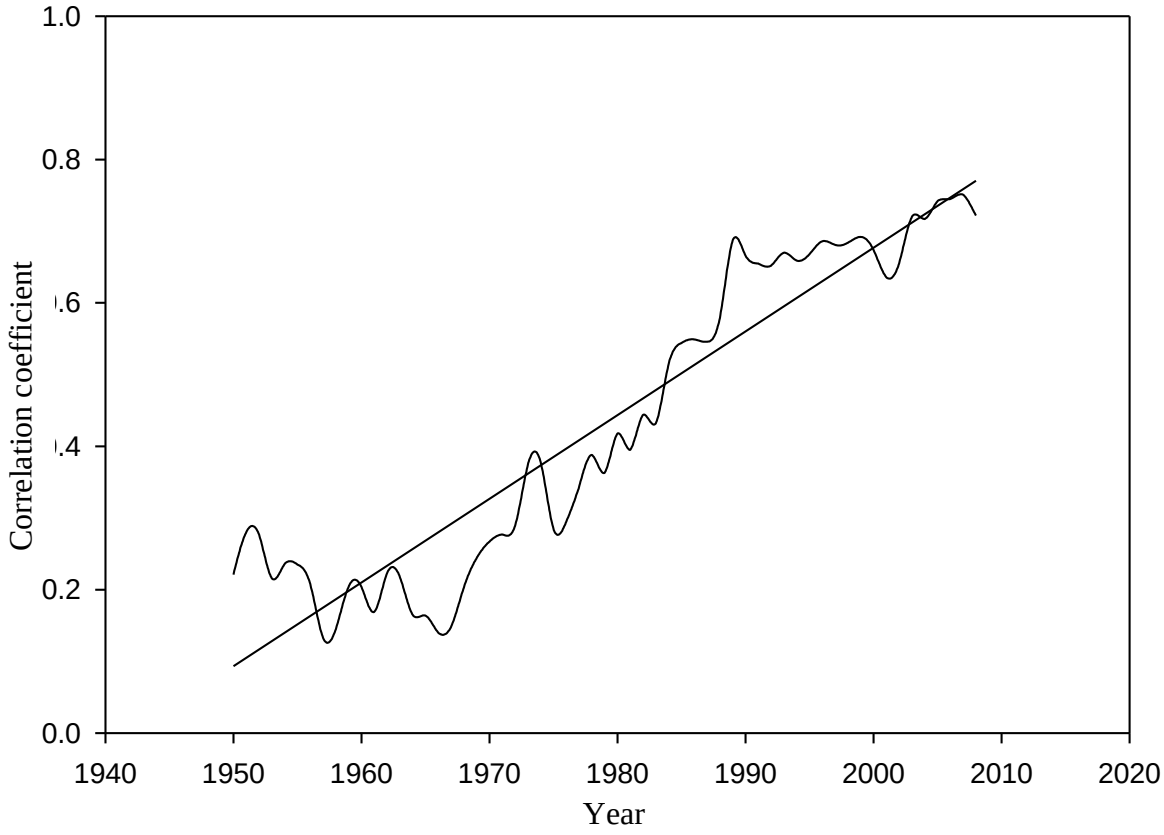

Figure 8. Regressed temporal change in July precipitation correlation coefficient (Y-axis) significance from 1918 to 2008 (X-axis) using 32-year moving intervals, Clemson Experimental Forest, SC, 2011. X-axis year marked 1950 represents the moving correlation coefficient by month for the period from 1918 to 1950.

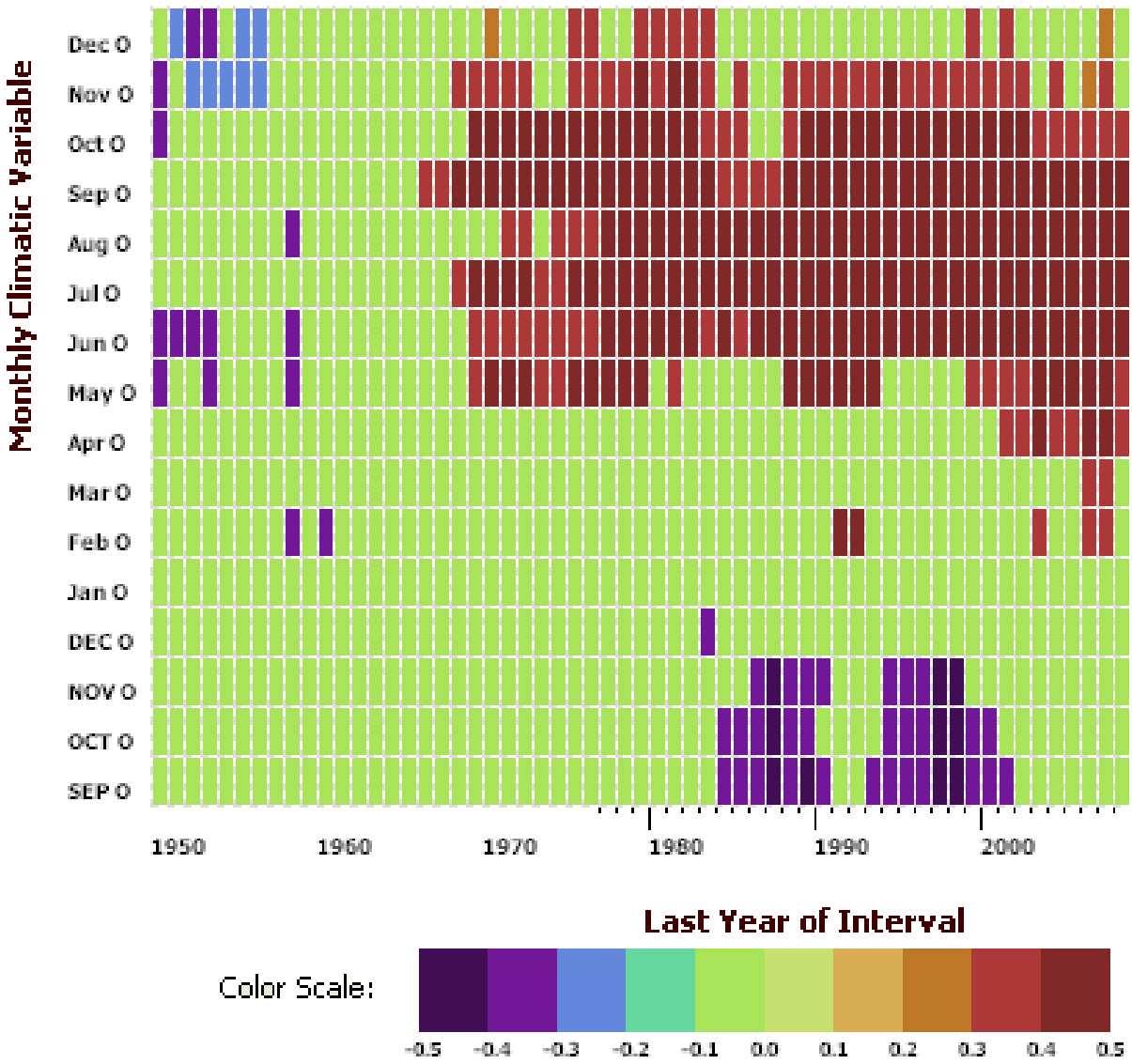

Figure 9. Moving correlation analysis between PDSI and the Clemson Experimental Forest loblolly pine chronology, using 32-year moving intervals from the period 1918 to 2008, SC, 2011. Last year of moving intervals are listed on the x-axis. Grid squares marked 1950 represent the moving correlation coefficient by month for the period from 1918 to 1950. Non-significant correlation intervals are shaded green, while significant correlations are colored according to the strength and direction of the correlation coefficient.

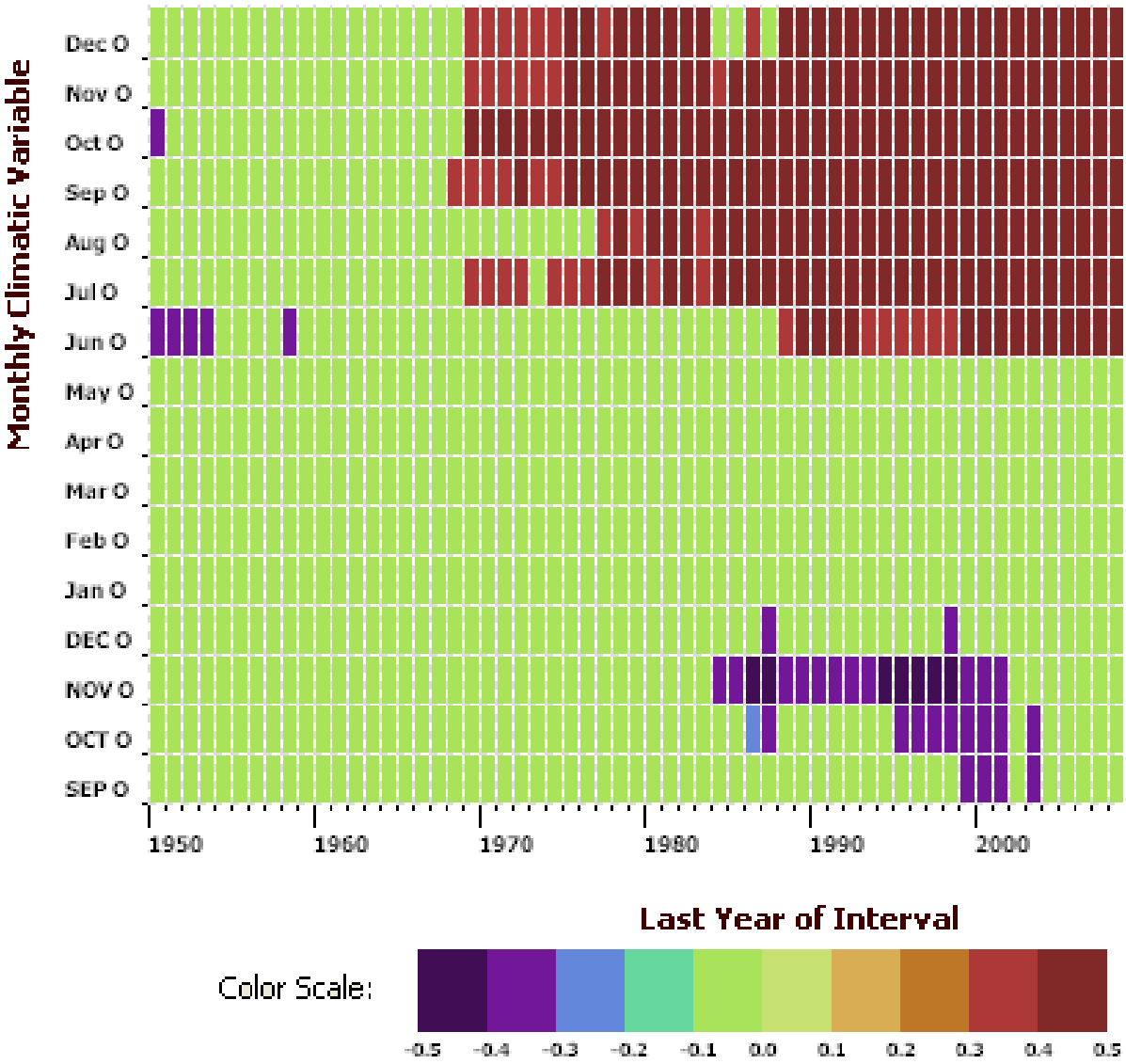

Figure 10. Moving correlation analysis between PHDI and the Clemson Experimental Forest loblolly pine chronology, using 32-year moving intervals from the period 1918 to 2008, SC, 2011. Last years of moving intervals are listed on the x-axis. Grid squares marked 1950 represent the moving correlation coefficient by month for the period from 1918 to 1950. Non-significant correlation intervals are shaded green, while significant correlations are colored according to the strength and direction of the correlation coefficient.

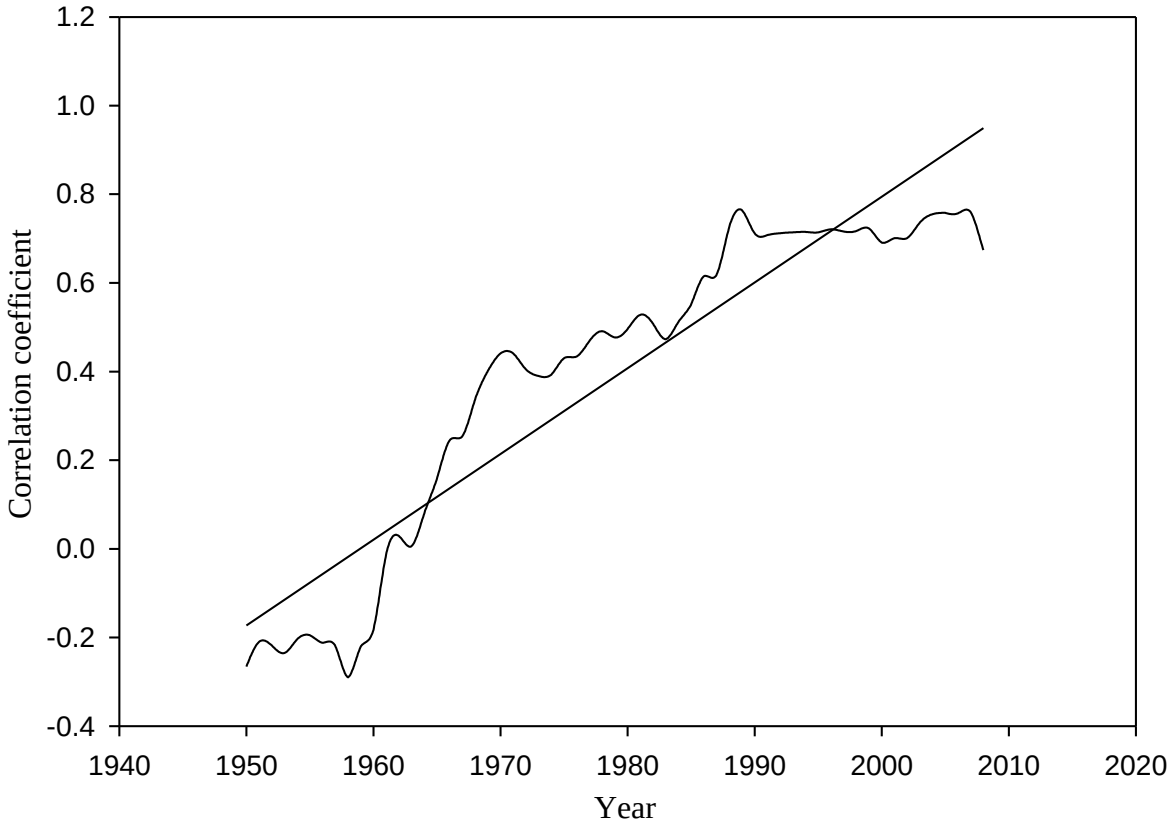

Figure 11. Regressed temporal change in July PDSI correlation coefficient (Y-axis) significance from 1918 to 2008 (X-axis) using 32-year moving intervals, Clemson, SC, 2011. X-axis year marked 1950 represents the moving correlation coefficient by month for the period from 1918 to 1950.

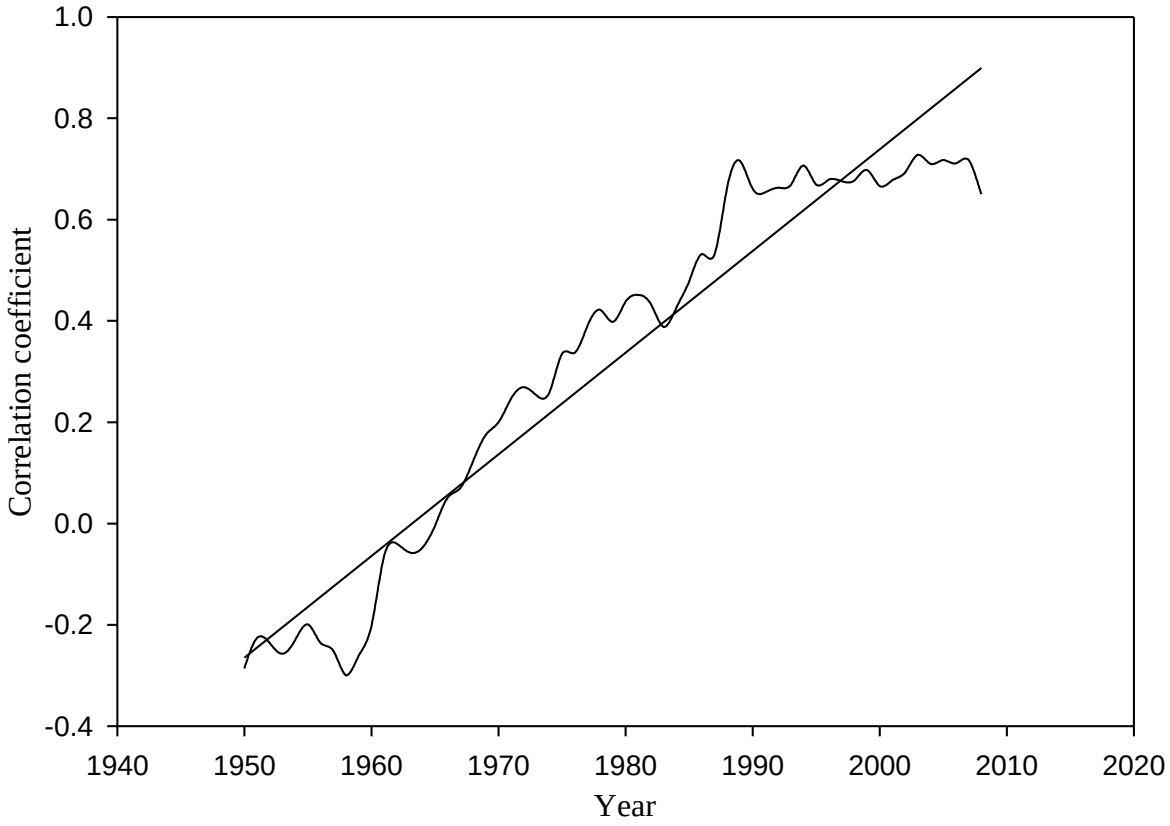

Figure 12. Regressed temporal change in August PHDI correlation coefficient (Y-axis) significance from 1918 to 2008 (X-axis) using 32-year moving intervals, Clemson Experimental Forest, SC, 2011. X-axis year marked 1950 represents the moving correlation coefficient by month for the period from 1918 to 1950.

Correlation Functions of Ocean-Atmospheric Oscillations

Correlation analyses suggested a complex relationship between ocean-atmospheric oscillations and tree growth, in addition to regional climate. Of the five variables analyzed (NAO, AMO, SOI, ENSO, and PDO indices), the master loblolly pine chronology was only correlated with NAO and PDO (Figure 13). Growth had a positive correlation with current NAO July ($r = 0.24$, $P < 0.05$) and September ($r = 0.27$, $P < 0.05$). This positive relationship suggests positive phases of NAO tended to coincide with increased tree growth. AMO was not significantly correlated with tree growth, suggesting sea surface temperatures of the Atlantic Ocean have a lower association than the standardized sea level pressure anomalies of the Atlantic Ocean.

The master chronology had an inverse relationship with the ocean-atmospheric oscillations in the Pacific Ocean than the Atlantic Ocean (Figure 13). Tree growth had a negative correlation with February ($r = –0.21$, $P < 0.05$), March ($r = –0.25$, $P < 0.05$), April ($r = –0.29$, $P < 0.05$), May ($r = –0.21$, $P < 0.05$), August ($r = –0.19$, $P < 0.05$), November ($r = –0.22$, $P < 0.05$), and December ($r = –0.28$, $P < 0.05$) PDO. The SOI and ENSO indices had no significant correlations with tree growth, suggesting air pressure and sea level pressure of the southern Pacific Ocean had minimal association with growth.

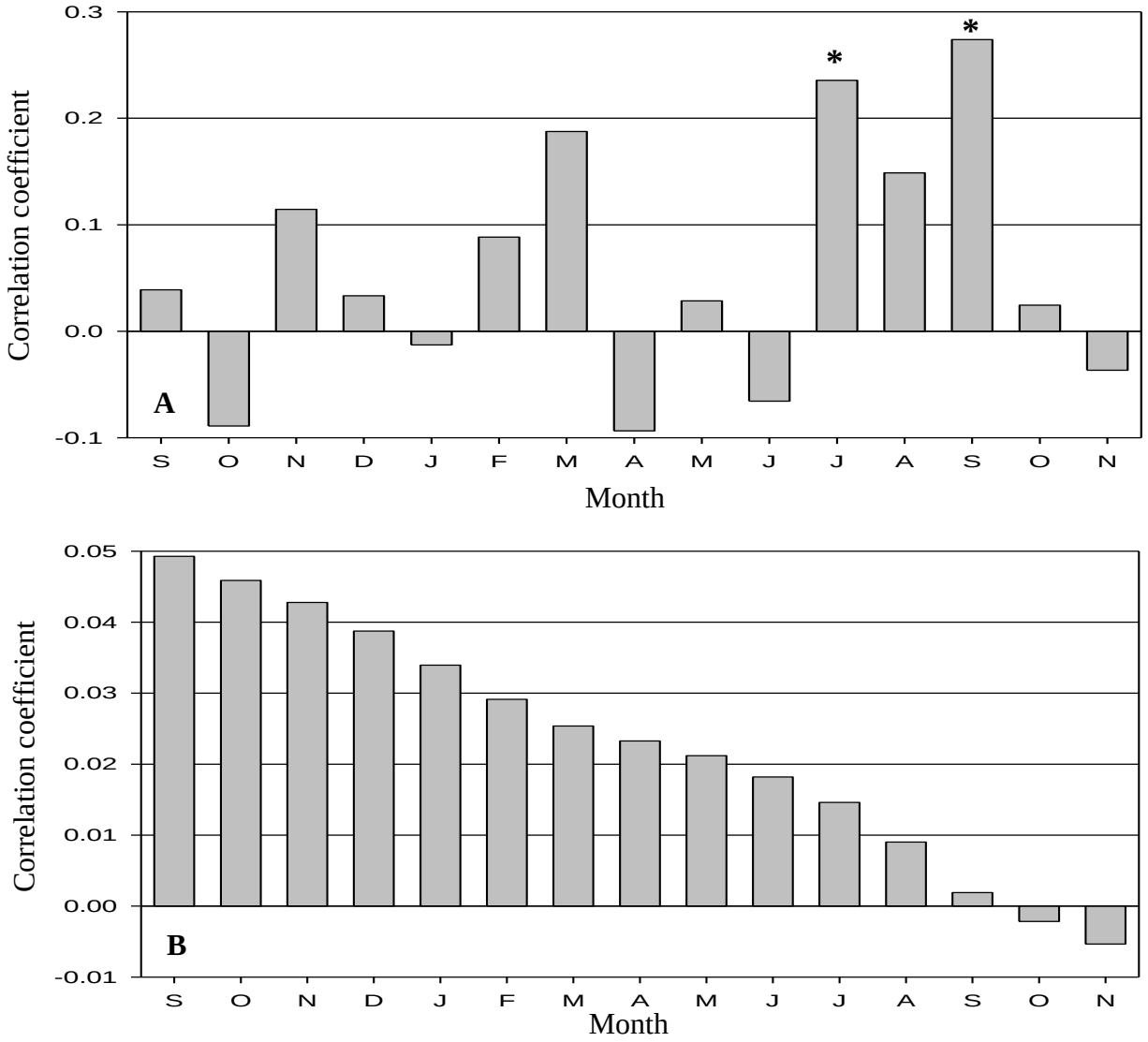

Figure 13. Correlation coefficients (Y-axis) of bootstrapped correlation function analyses between the Clemson Experimental Forest loblolly pine chronology and NAO (A), AMO (B), SOI (C), ENSO (D), and PDO (E) from the previous September to the current November (X-axis) from the period 1918 to 2008, Clemson, SC, 2011. Asterisks (*) indicates significance at α = 0.05.

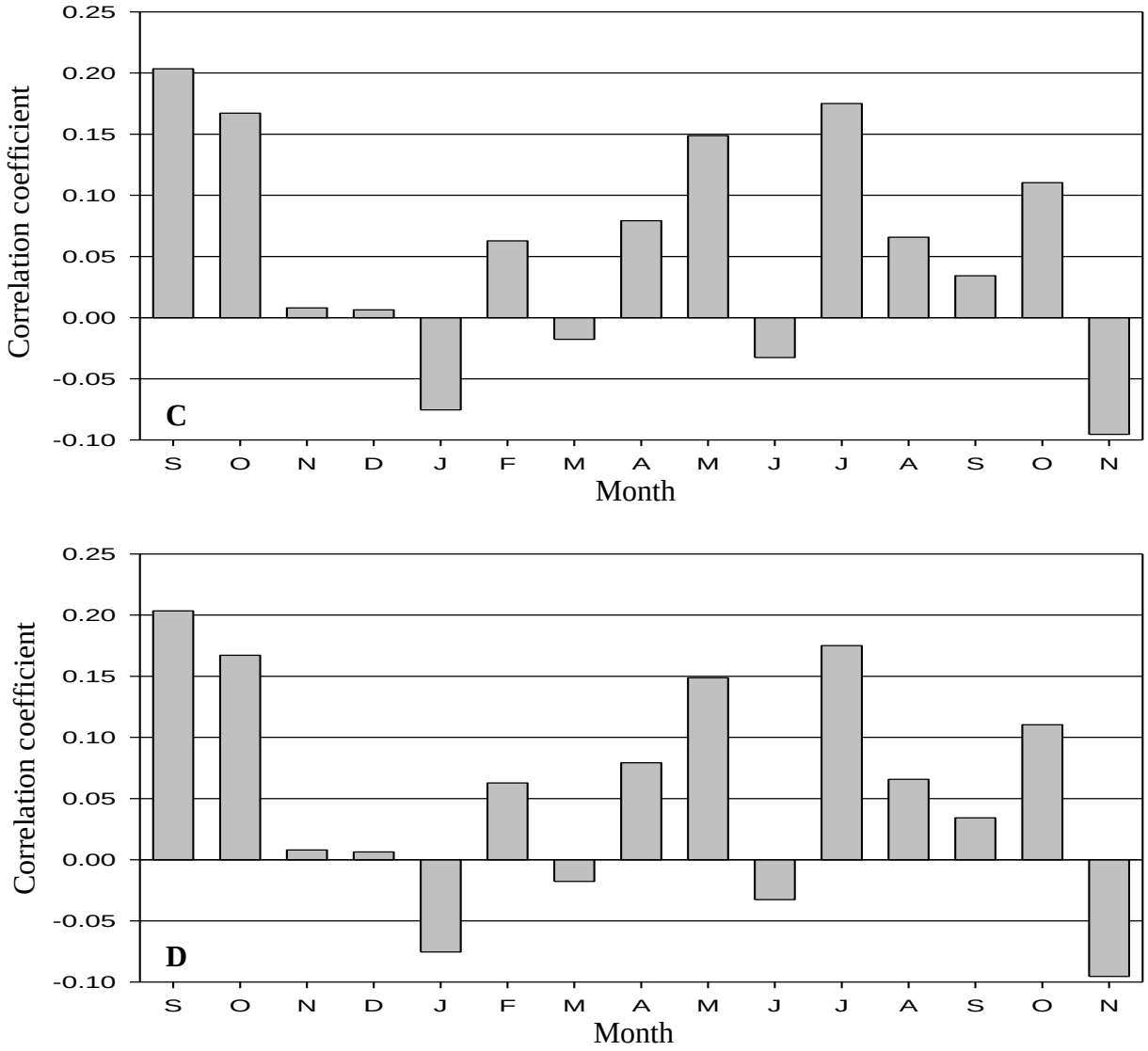

Figure 13 (Continued). Correlation coefficients (Y-axis) of bootstrapped correlation function analyses between the Clemson Experimental Forest loblolly pine chronology and NAO (A), AMO (B), SOI (C), ENSO (D), and PDO (E) from the previous September to the current November (X-axis) from the period 1918 to 2008, Clemson, SC, 2011. Asterisks (*) indicates significance at $\alpha = 0.05$.

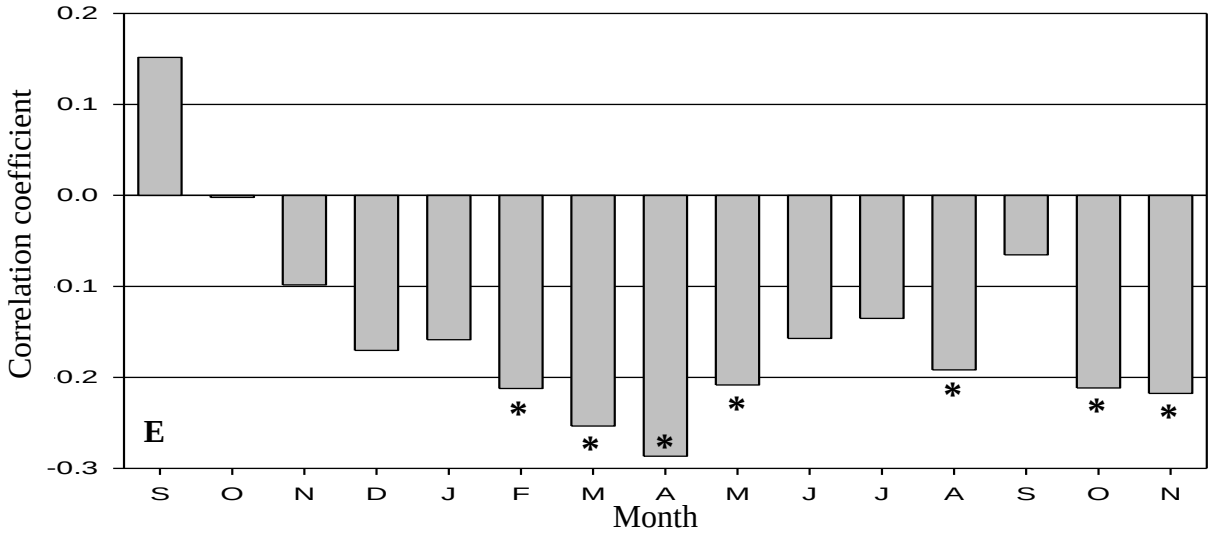

Figure 13 (Continued). Correlation coefficients (Y-axis) of bootstrapped correlation function analyses between the Clemson Experimental Forest loblolly pine chronology and NAO (A), AMO (B), SOI (C), ENSO (D), and PDO (E) from the previous September to the current November (X-axis) from the period 1918 to 2008, Clemson, SC, 2011. Asterisk (*) indicates significance at α = 0.05.

Moving Correlation Analysis of Ocean-Atmospheric Oscillations

Moving correlation analyses suggest NAO and PDO have cyclical influences on tree growth. NAO exhibits a positive correlation for July beginning in the period 1957 to 1988 (Figure 14). Since then, NAO has been in a positive phase. In the eastern US, the positive phase of NAO is typically associated with mild and wet winters, but also influences hurricanes. A positive July correlation suggests the positive phase of NAO influences precipitation, potentially the result of increased hurricane activity and summer storms.

PDO exhibits cyclical negative correlations with tree growth for current spring, most of the summer, and winter associated with the warm phase of PDO. The 1918 through 1963 and the 1950 through 2000 had negative correlations, while the period between showed no relationships, which coincides with the warm and cool phases of PDO (Figure 15). In the southeastern US, warm phases of PDO are associated with below average temperatures. Lower than average temperatures, particularly in drought stressed trees, tends to increase tree growth because there is less soil moisture loss combined with increased transpiration and photosynthesis.

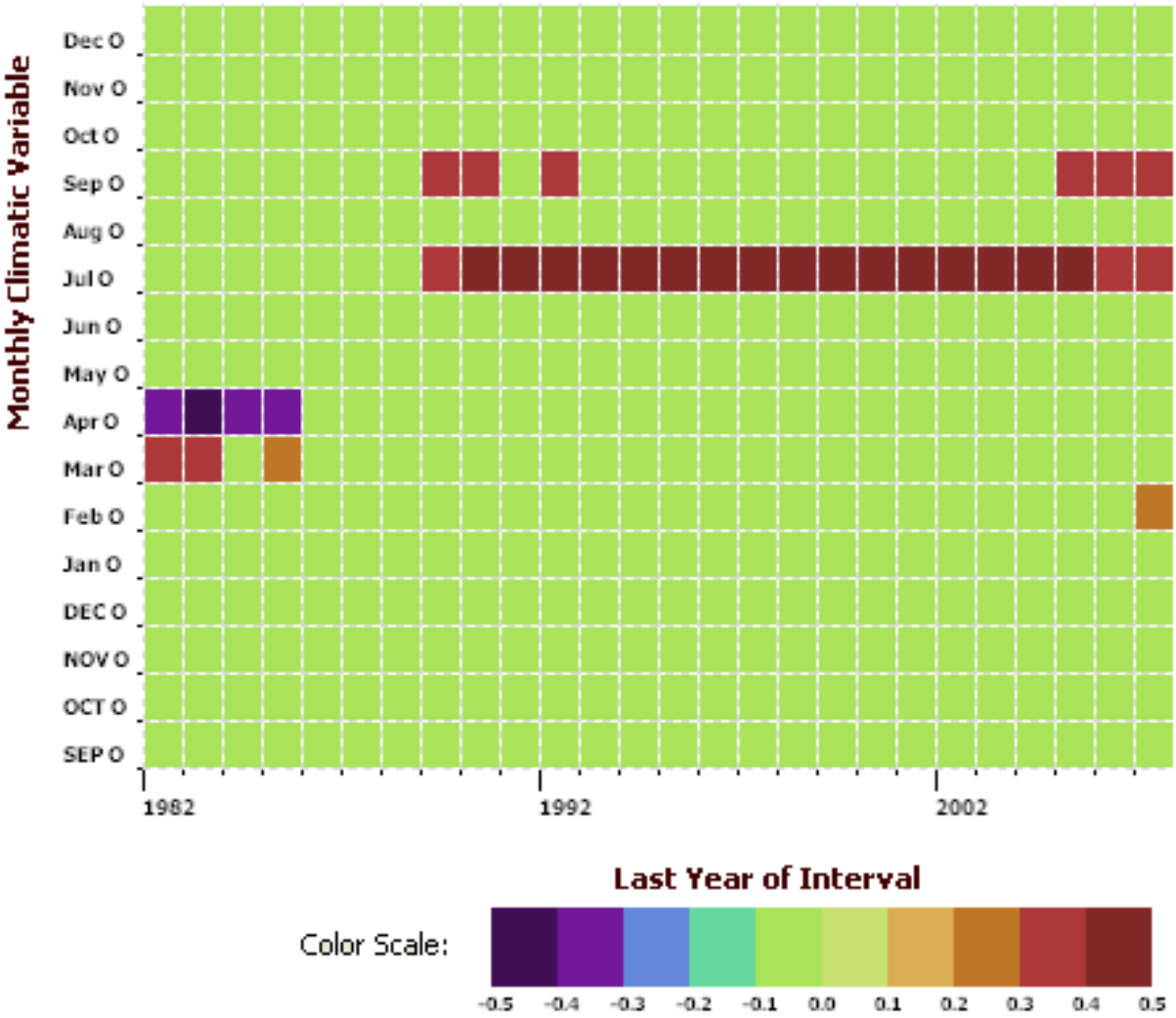

Figure 14. Moving correlation analysis between NAO and the Clemson Experimental Forest loblolly pine chronology, using 32-year moving intervals from the period 1950 to 2007, SC, 2011. Last years of moving intervals are listed on the x-axis. Grid squares marked 1950 represent the moving correlation coefficient by month for the period from 1950 to 1982. Non-significant correlation intervals are shaded green, while significant correlations are colored according to the strength and direction of the correlation coefficient.

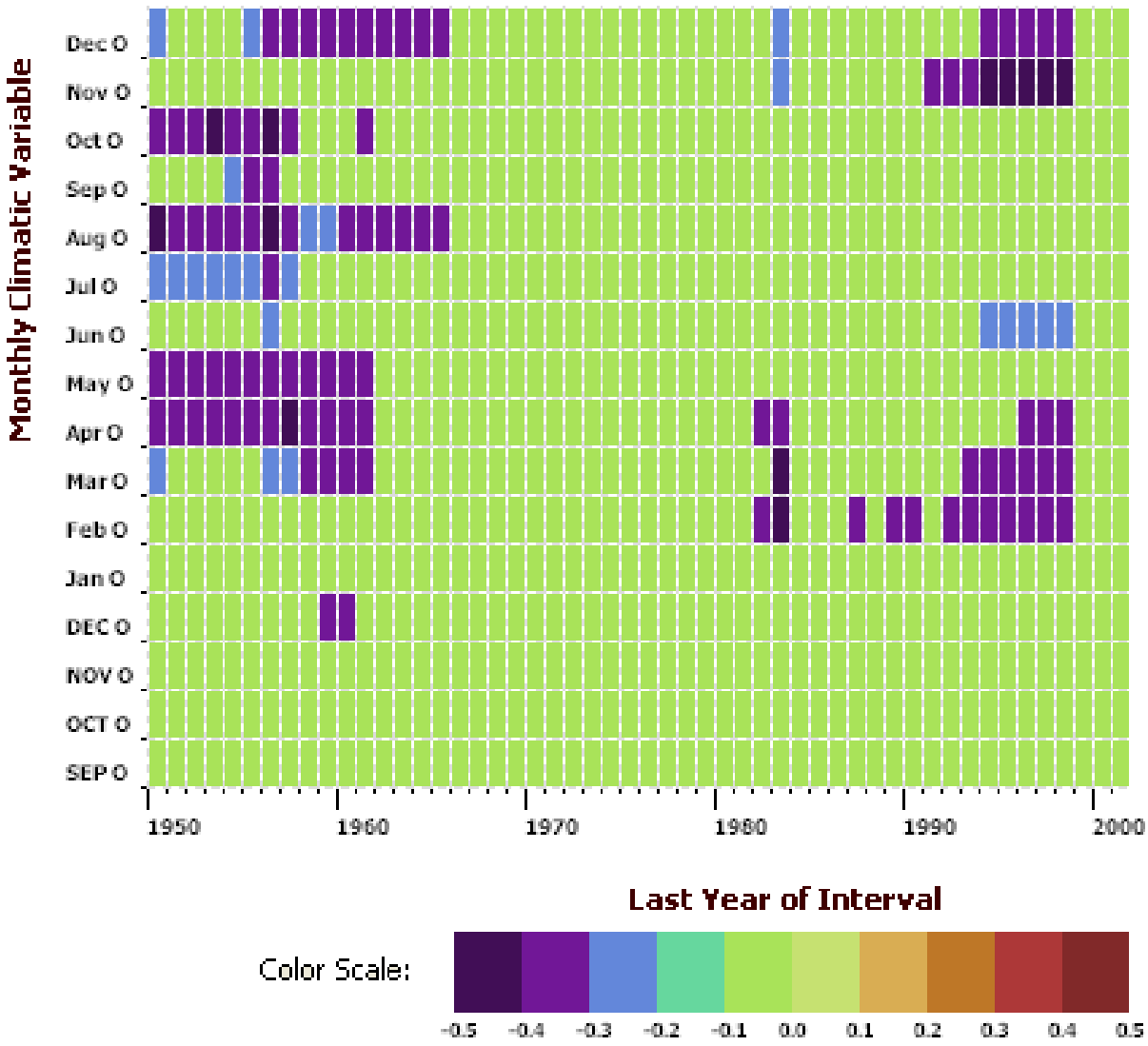

Figure 15. Moving correlation analysis between PDO and the Clemson Experimental Forest loblolly pine chronology, using 32-year moving intervals from the period 1918 to 2001, SC, 2011. Last years of moving intervals are listed on the x-axis. Grid squares marked 1950 represent the moving correlation coefficient by month for the period from 1918 to 1950. Non-significant correlation intervals are shaded green, while significant correlations are colored according to the strength and direction of the correlation coefficient.

CHAPTER IX
DISCUSSION FOR GROWTH-CLIMATE RESPONSE OF COASTAL PLAIN PLANTATION LOBLOLLY PINE, CLEMSON EXPERIMENTAL FOREST, SC, USA

Loblolly Pine Climate-Growth Relationships

Despite being plantation loblolly pine, the Clemson Experimental Forest chronologies showed strong climate and ocean-atmospheric oscillation responses. Based on other studies of loblolly pine in the Southeast and studies of other yellow pines in the Piedmont, responses to dormant season and/or prior summer precipitation, early and late growing season temperatures, and mid-summer and/or late-summer drought sensitivity were expected, but no significant ocean-atmospheric oscillation responses were expected (Cook et al., 1998; Henderson and Grissino-Mayer, 2009; Grissino-Mayer et al., 1989; Copenheaver et al., 2002; Cleaveland, 1975). This network of chronologies exhibited climate responses to dormant season precipitation, early growing season temperature, current year and previous year late growing season temperature, and late-summer drought sensitivity. However, the strongest climate responses were exhibited by July precipitation, temperature, and PDSI. In addition to climate responses, this tree-ring data also exhibited cyclical ocean-atmospheric oscillation responses to late summer NAO and dormant through growing season PDO.

Radial Growth Response to Temperature and Precipitation

Temperature and precipitation had multiple influences on Piedmont loblolly pine growth in this study, including early and late in the current year growing season, during the end of the previous growing season, and when trees are potentially most stressed in the current summer, similar to findings by Grissino-Mayer et al. (1989), Cook et al. (1998), and Cleaveland (1975). Higher than average temperatures in March combined with increased February and end of the previous growing season precipitation resulted in increased early growing season growth. Higher than average November temperatures appeared to increase growth and potentially delayed dormancy (Perry, 1971). Although Cook et al. (1998) only found weak or non-significant growth-climate relationships, the moving correlation analyses suggest a temporal shift toward stronger relationships that were not evident at the time their study was conducted.

Similar to Grissino-Mayer et al. (1989), this study found a strong negative temperature relationship and a strong positive precipitation relationship in July, while Cook et al. (1998) found no late summer relationships. Cleaveland (1975) found growth was positively related to temperature until July then an inverse relationship in subsequent month, with a positive growth-precipitation relationship in Piedmont region shortleaf

pine. These differences in the climate-growth relationships are likely due to species differences or differences in calculation methodology, specifically the use of bootstrapping techniques to identify significance, because the moving correlation analyses did not identify the same climate-growth relationships patterns to Cleaveland (1975) prior to 1975.

Radial Growth Response to Drought Sensitivity

Radial growth responses to PDSI and PHDI were similar and longer lasting than temperature and precipitation responses. Minor differences occurred between PDSI and PHDI responses resulting from the lag associated with hydrologic processes. However, PHDI showed a more constant response than PDSI. Both PDSI and PHDI had a strong positive relationship with late summer and fall growth. High positive values of PDSI and PHDI indicate low drought stress and abundant soil moisture. When combined with the strong July temperature and precipitation signals, the strong positive PDSI and PHDI relationships with late summer and fall growth indicated strong drought sensitivity.

Late summer is the most drought stressful period for loblolly pine in the Piedmont region of South Carolina, as with much of the Southeast. Although Cleaveland (1975) did not examine the influence of drought indices on shortleaf pine growth, the late-summer and fall negative temperature/positive precipitation relationships reported are indicative of drought sensitivity. Henderson and Grissino-Mayer (2009) also found high correlations between PDSI and longleaf pine growth from July to November. Drought sensitivity was not readily evident in loblolly pine in the easterly locations of the Southeast (Cook et al., 1998), but changes in the temporal stability of the relationship are likely the result of the inconsistencies between studies.

Radial Growth Responses to Ocean-Atmospheric Oscillations

Ocean-atmospheric oscillations did not have relationships as strong as the climate variables evaluated in the study. The ocean-atmospheric oscillations that significantly influenced growth, NAO and PDO, are generally associated with influencing winter weather patterns. However, evidence from the study and others suggests NAO and PDO also influence summer weather (Nigram et al., 1999).

NAO controls the strength and direction of westerly winds and storm tracks across the North Atlantic and is associated with the Arctic oscillation. Resulting fluctuations in the atmospheric pressure at sea level in the North Atlantic have strong influences on the jet stream. In addition to influencing winter weather, these oscillations also influence hurricane tracks and summer precipitation. The chronology only provides enough data to evaluate one cycle of NAO, but the results suggest a potential relationship between increased summer growth and the positive phase of NAO. During the positive phase of

NAO, the Southeast generally has cooler than average temperatures and wetter than average precipitation; which increases growth during the months that are typically the most drought-stressed.

PDO is similar to a long-lived El Niño-like pattern of Pacific climate variability which persists on average 20 to 30 years (Zhang et al., 1997). Warm and cool phases of PDO are associated with warmer or cooler than average sea surface temperatures in the Pacific Ocean. Changes in sea surface temperatures result in large-scale temperature and precipitation shifts across North America. Although the chronology only provides enough data to evaluate one cycle of PDO, the results suggest a potential relationship between decreased growth and the warm phase of PDO. Tree-growth decreases because warm phases of PDO result in below average October through March temperatures and below average precipitation from May to September for the Southeast. The lower than average temperatures at the beginning and end of the growing seasons potentially shorten the growing season or slows growth. In addition, the lower average summer precipitation results in decreased growth during the summer due to increased drought stress.

Temporal Consistency of Climate-Growth Relationships

Moving correlation analyses revealed some inconsistencies in the climate-tree growth relationships over time. All of the significant climate variables identified in the correlation and response function analyses have strengthened through time. The temperature relationship has varied through time, with both positive and negative correlations with tree growth for differing months. Beginning in the period 1918 to 1950, May temperatures predominantly influenced growth, but the strength of the relationship decreased and was no longer significant by the period 1931 to 1963. In the period 1931 to 1963, previous October temperatures had an inverse relationship with growth, which increased in strength until the period 1926 to 1958 then decreased and was no longer significant by the period 1945 to 1977. Since the 1945 to 1977 period, an inverse July temperature relationship has developed and increased in strength. However, the recent trend, the period 1968 to 2000, suggests the relationship may be weakening.

The result of a combination of phase shifts in ocean-atmospheric oscillations and the changes in the temporal consistency of the temperature-growth relationship cannot be attributed to any single ocean-atmospheric oscillation for the entire length of the chronology. However, the most recent warm phase of PDO that began in the late 1970s is likely the cause of the inverse relationship between July temperature and tree growth. Although warm phases of PDO result in below average rainfall, when temperatures are lower than average there is less drought stress and trees can increase transpiration.

Although possible, the likelihood of the changes in the relationships due to stand dynamics influences or physiological influences is low. Immature trees do not tend to be growth limited, and as a result climate-growth relationships are not evident. To alleviate

this influence, mature trees were used and the early growth was excluded (approximately the first 10 years depending on the sample). The influence of stand dynamics is likely minimal because there is no evidence in the growth chronology of disturbance events, such as the 2006 ice storm, the 2002-2003 southern pine beetle attack, or any of the prescribed fires.

The precipitation-tree growth relationship also varied through time and shifted from negative correlations to positive correlations. In period 1919 to 1950, there was a negative correlation between October precipitation and tree growth, but by the 1929 to 1960 period the relationship degraded and was no longer significant. For the next decade, a positive September and short term, likely erroneous, positive relationships occurred throughout the growing season. Short term relationships are erroneous because they do not consistently respond to climate signals and thus are likely confounded by other environmental variables. By the period 1939 to 1970, positive correlation relationships developed between precipitation and July and February. Although both relationships have increased in strength through time, the July correlations are nearly twice the February correlations.

Similar to the temperature-tree growth relationships, the changes in the temporal consistency of the precipitation-tree growth relationship cannot be attributed to any single ocean-atmospheric oscillation. Shifting relationships are likely the result of a combination of phase shifts in ocean-atmospheric oscillations. The most recent relationship is likely the result of the shift to the positive phase of NAO, with cooler than average temperatures and wetter than average precipitation.

Unlike temperature and precipitation, PDSI and PHDI had few erroneous relationships prior to the 1934 to 1965 period. Since the 1934 to 1965 period, both PDSI and PHDI have had strong positive correlations with all growing season months from May through November. The positive correlations have increased steadily since the 1960s suggesting the trees are becoming more drought-sensitive. Around 1960, AMO shifted from a warm to a cool phase and lasted through the 1990s (Gray et al. 2004). Although AMO was not a significant oscillation alone, this shift coincides with the PDSI- and PHDI-tree growth relationships in the chronologies, suggesting it has an influence over that period. However, since the 1990s, AMO has started to shift toward a cool phase associated with increased precipitation, yet the climate-tree growth relationship continues to increase in strength, suggesting an additional influence.

In addition, a negative relationship between tree growth and the previous year September through November occurred from the 1954 to 1985 period to the 1971 to 2002 period. This negative relationship suggests as drought in the previous year increases, tree growth in the following year is decreased. This relationship corresponds with a period in which PDO and AMO are simultaneously positive, an occurrence with often leads to drought and decreased tree growth (McCabe et al., 2004).

Evidence of NAO in the chronology did not become evident until the period 1958 to 1989. This period coincides with the positive phase shift of NAO. Because the NAO dataset used in the analyses begins at 1950, only the positive phase and the end of the negative phase of NAO in 1982 were used. Some evidence exists of a potential negative relationship between April negative phase NAO, but without a longer NAO dataset the nature of the relationship is currently speculative. NAO is typically associated with winter weather, but the results suggest a strong positive correlation with July tree growth in the positive phase, resulting from cooler than average temperatures and wetter than average precipitation. The latter correlations appear to be decreasing suggesting NAO may be shifting phases.

The relationship between PDO and tree growth was not constant through time nor through the full PDO cycle. The lack of consistency through the PDO cycle is likely due to the influence of ENSO or other oscillation interactions. ENSO's quasi-periodic climate pattern occurs on a 5- to 7-year cycle and influences temperatures, wind patterns, and precipitation across North America. The ENSO North American climate oscillations are strongly dependent on the phases of PDO (Newman et al., 2003). Although ENSO was not significantly correlated with tree growth, it had a similar pattern to PDO, in that it was negatively correlated with tree growth during the growing season. When ENSO and PDO are "in phase" more extreme anomalies typically occur. When compared with the phases of ENSO, the moving correlations analyses concur with this pattern suggesting tree growth is most significantly influenced when PDO and ENSO are "in phase."

CHAPTER X
CONCLUSIONS

Fire Signature

A nutrient fire signature can neither be confirmed nor denied. The research suggests the potential for a nutrient signature, particularly in the immobile nutrients, but further investigation is required. The methodology and technique used in this study confirmed that ICP-MS can be used to identify differences in mean nutrient concentrations, but not to identify a fire signature using multiple year resolution. Future studies should consider finer resolution, other techniques, and site differences. Examination at annual resolution could potentially determine if a nutrient gradient is present, and its duration. Other techniques, particularly spectroscopy or electron microscopy, may prove to be more efficient and accurate with regard to ease of sampling and sample preparation than ICP-MS. Spectroscopic techniques and electron microscopy are minimally destructive when compared to ICP-MS, and require substantially smaller sample quantities to perform.

Differences in the potential fire signature may occur due to soil, vegetation composition, fire intensity, soil microbial community, and climate differences. This study only focused on the potential for a fire signature in sapwood. Speculation on the potential fire signature present in heartwood is not discussed because there is a large flux of ions related to heartwood formation that is not comparable to sapwood chemistry. The multiple year-periods used in this study were found to be unsuitable for climate analysis. However, given annual resolution of nutrient concentrations and radial growth data, climate influences could be identified and analyzed.

Climate

The results suggest, contrary to the Cook et al. (1998) study, that climate plays an important role in the growth of mature loblolly pine in the South Carolina Piedmont. Loblolly pine in the South Carolina Piedmont is highly drought sensitive in the summer and is thus also sensitive to summer temperatures and precipitation. Temporal consistency results suggest drought sensitivity increases with tree age. Unlike the climate variables, the relationships between ocean-atmospheric oscillations and radial growth are more cyclical in nature, and only appear to influence growth in positive phases. However, without a longer series chronology, the long-term relationships cannot be fully explored.

Temperature, precipitation, PDSI, positive phase NAO, and positive phase PDO are the primary drivers of loblolly pine radial growth for their respective seasons. Increased spring temperatures and precipitation positively influence spring radial growth. Summer growth is influenced by PDSI and the positive phase of NAO. High PDSI values in the summer increases summer growth due to greater moisture. Summer temperatures decrease during the positive phase of NAO, while increasing precipitation also increases

summer growth. Radial growth decreases with the positive phase of PDO because decreased fall and winter temperatures shorten the growing season and minimize favorable conditions for photosynthesis. Because loblolly needles are retained year round, photosynthesis and growth can occur when temperatures are favorable, especially in the early spring and late fall. In contrast, deciduous trees lose their foliage and are triggered by photoperiod, so growth is limited to when foliage is present. Thus, an extended growing season for conifers is an opportunity for more days of pine growth.

The long-term cycles of PDO and NAO are not as likely to influence pine management as spring temperature, precipitation, and PDSI because pine has short rotation lengths, typically less than one oscillation cycle. Temperature, precipitation, and PDSI can influence timing of site preparation and intermediate operations, fire weather, and planting success. The positive phase of the PDO cycle potentially influences timing and success of planting, indicating planting during this phase can be conducted earlier than normal to allow more time for seedling establishment before winter.

Intensive silvicultural management in pines represses much of the climate signal because trees remain growing vigorously and experience minimal stress. Although nutrient concentrations could not be quantified to determine if they relate to the climate-tree growth relationship in this study, nutrients are assumed to play an important role in the relationship. Without a growth-limiting factor, such as nutrients, climate-growth analyses are not possible because interannual variation is minimal and series are complacent. Soils throughout the Southeast are highly weathered and nutrient poor (Buol et al., 1997). Since loblolly pine in the South Carolina Piedmont experiences interannual variability that does not have a consistent growth response to any climate or oscillation variable, climate is not the most limiting factor in these stands. The soils results in this study further support these findings and suggest nutrient management in loblolly pine is essential to maintain growth and vigor of stands, so that climatic influences can be minimized.

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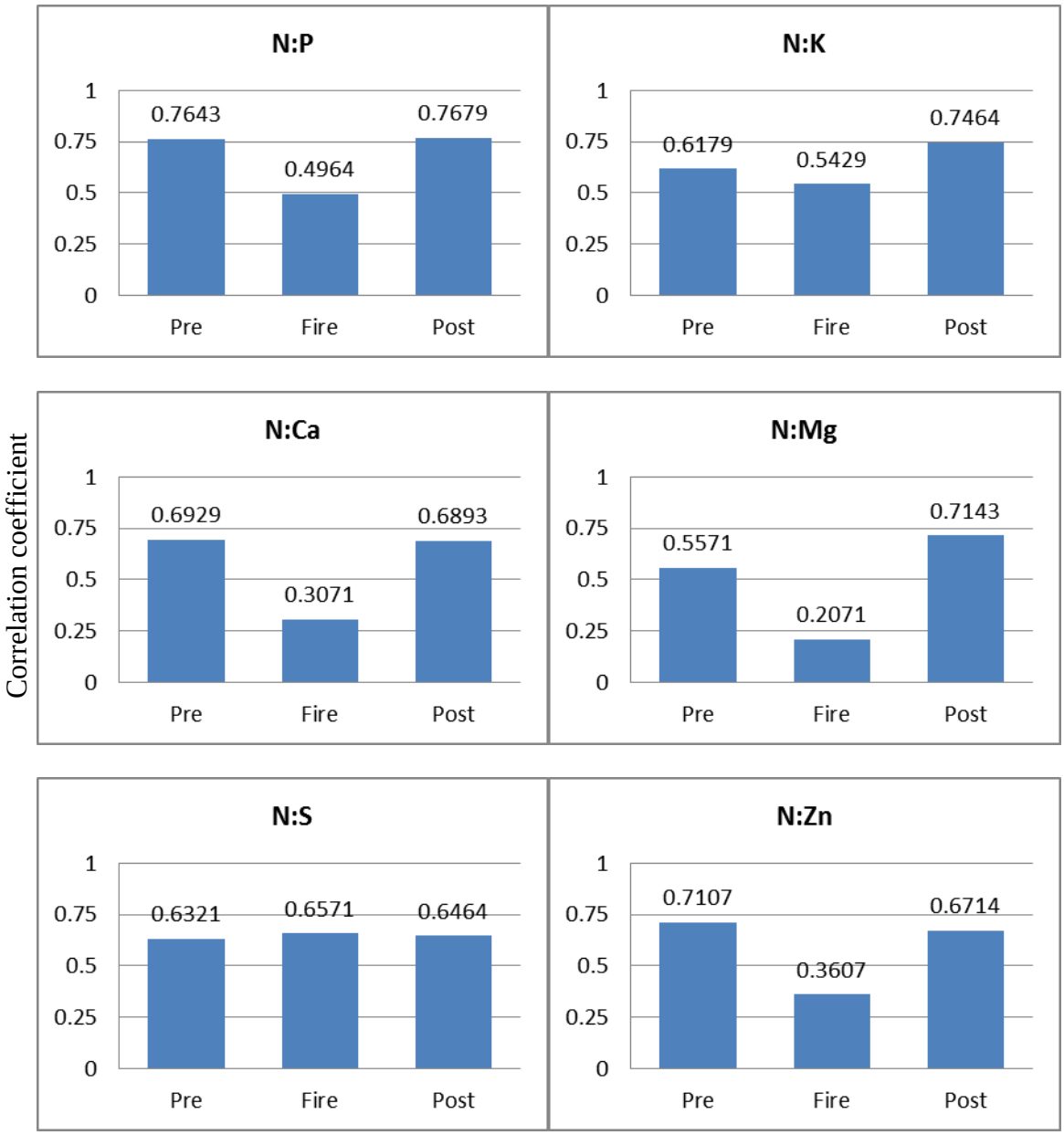
APPENDIX

Spearman correlation coefficients for Piedmont loblolly pine xylem nutrient concentrations by period, Clemson Experimental Forest, SC, 2011.



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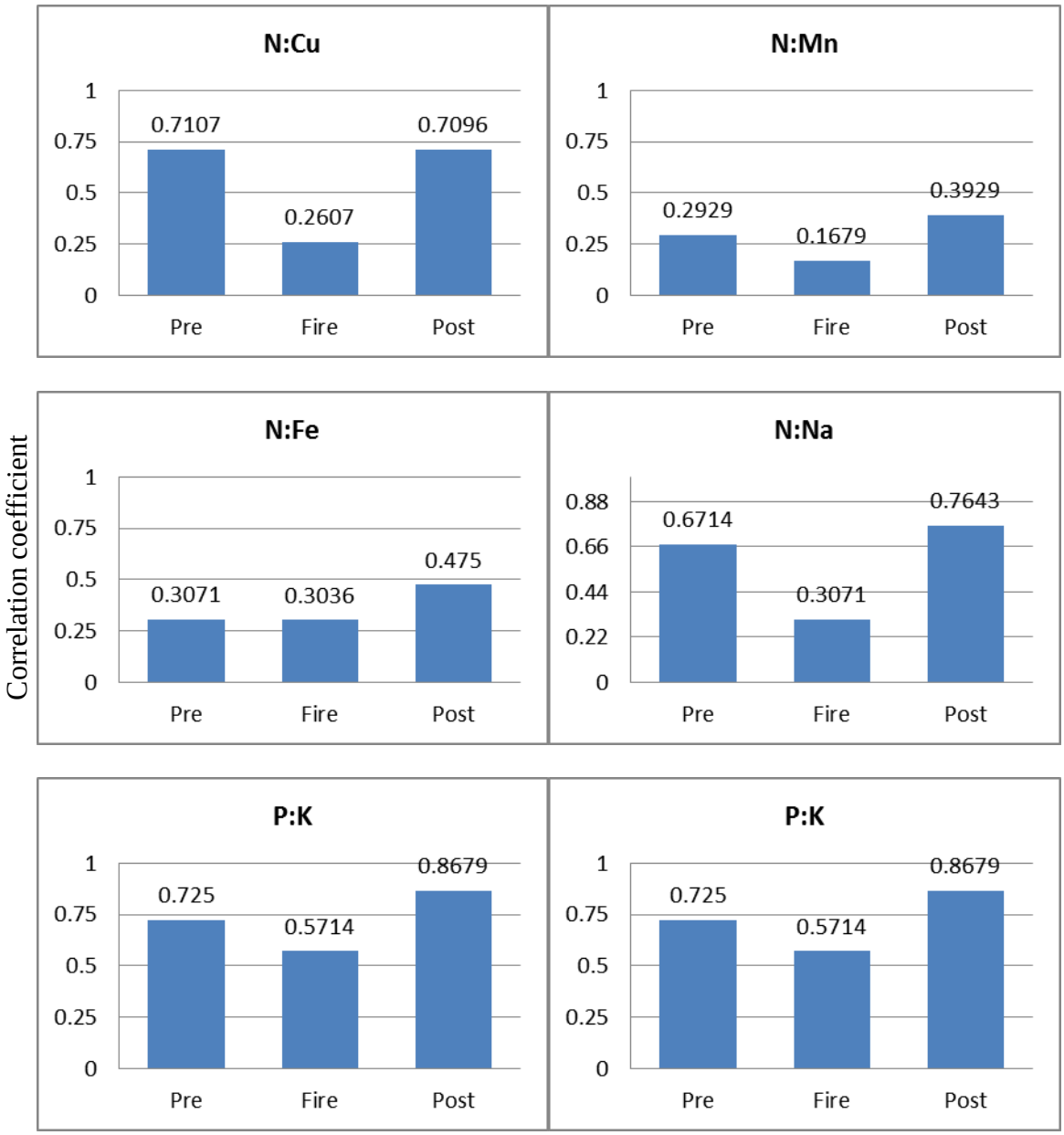

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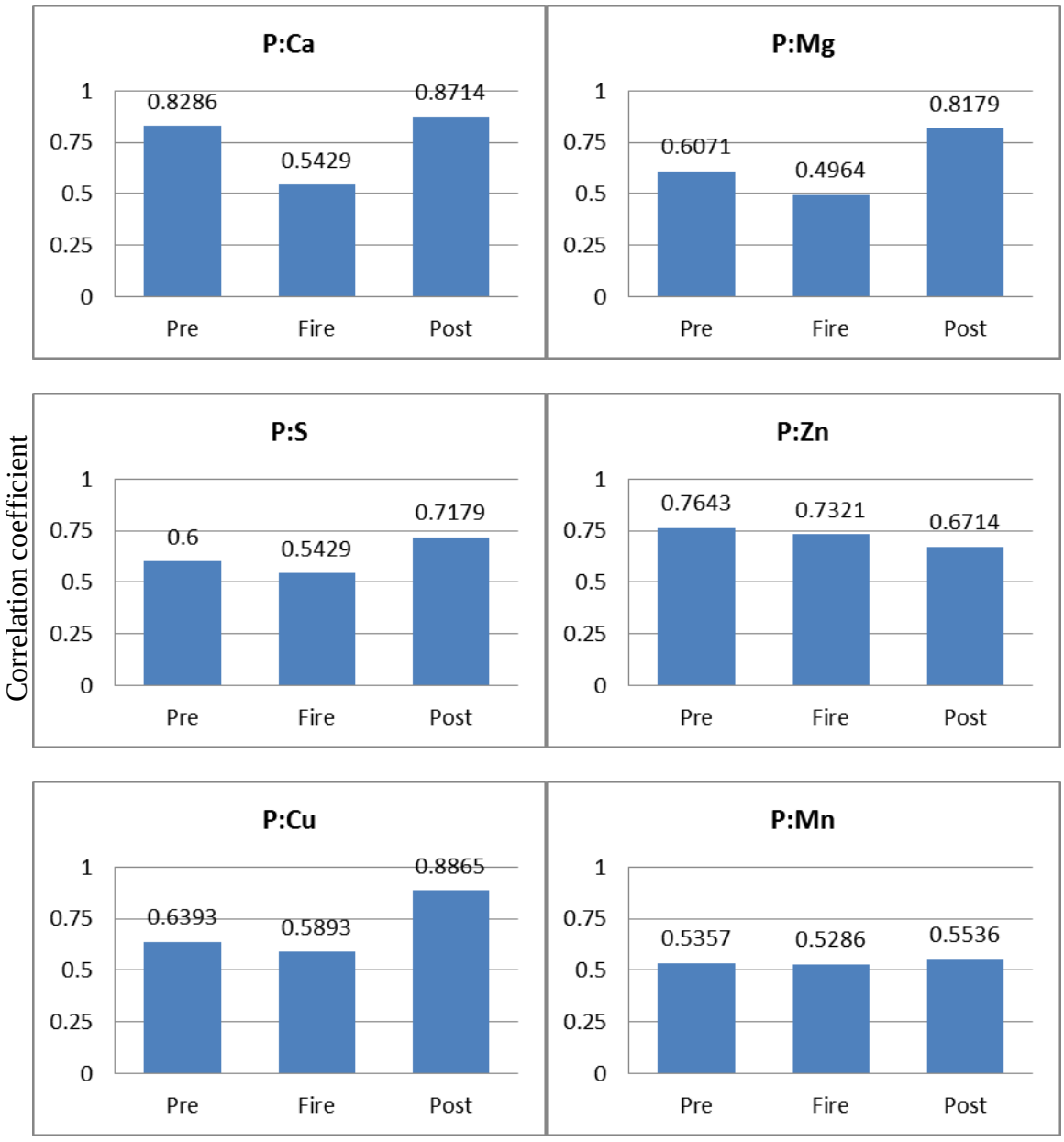

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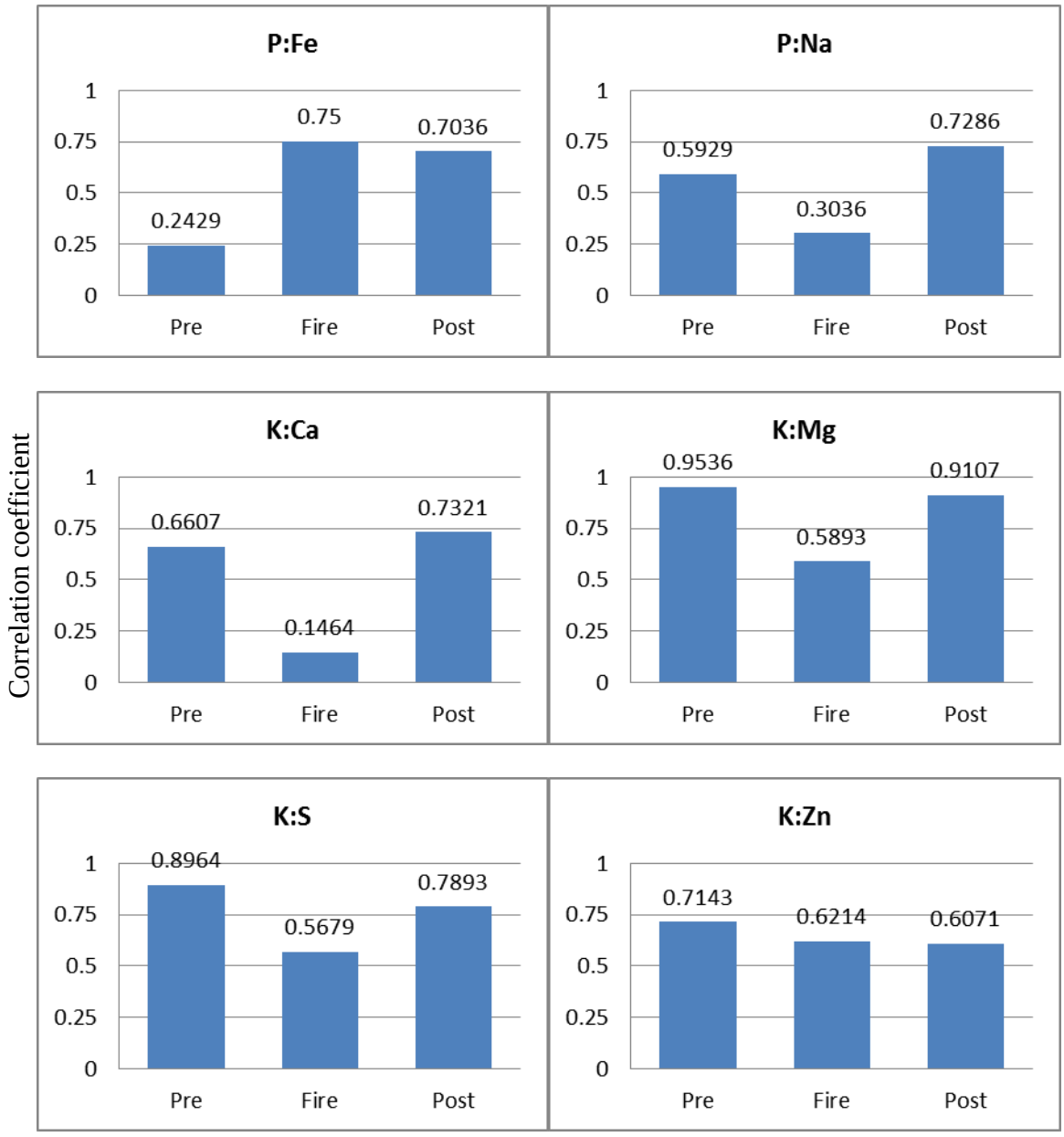

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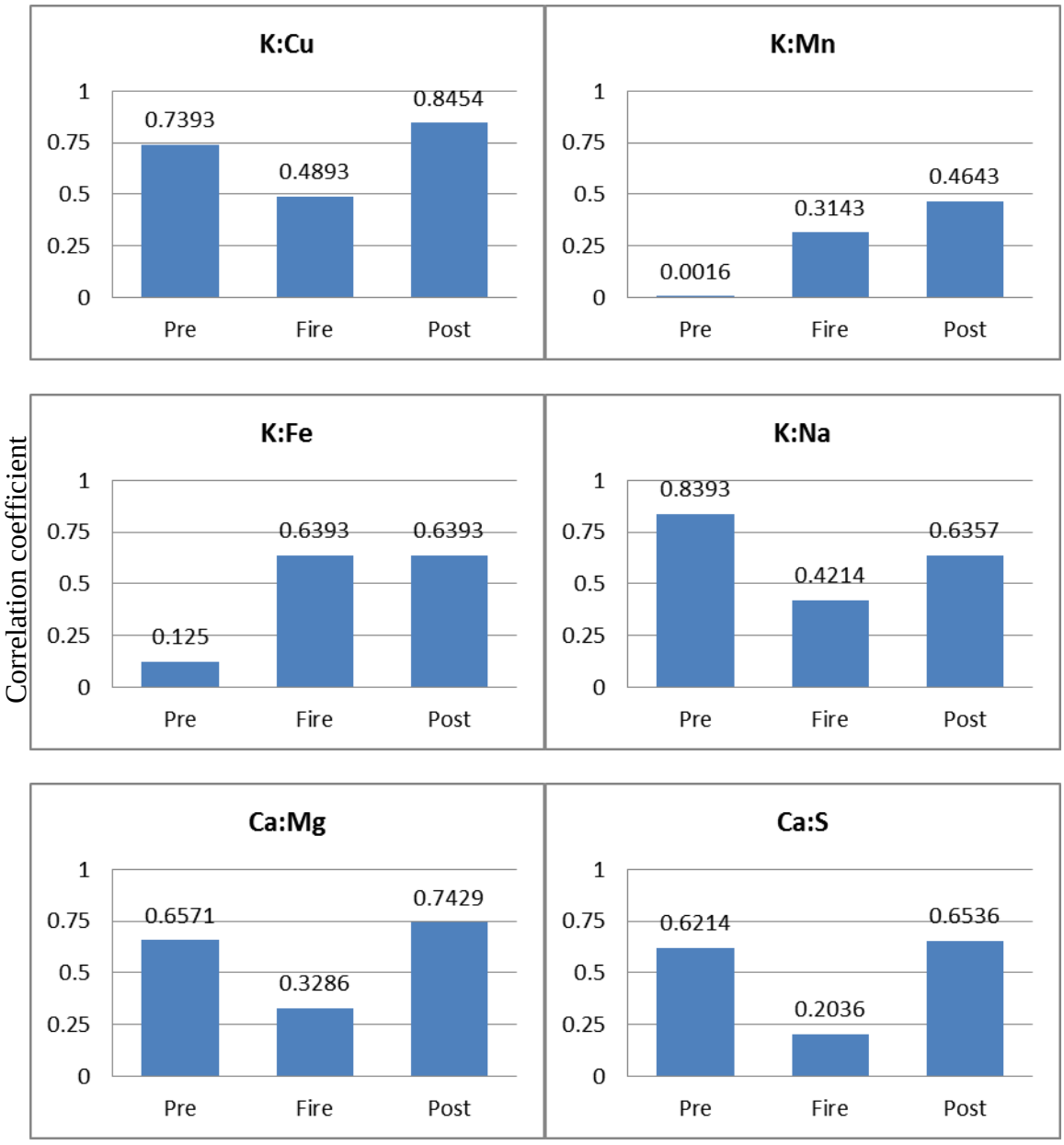

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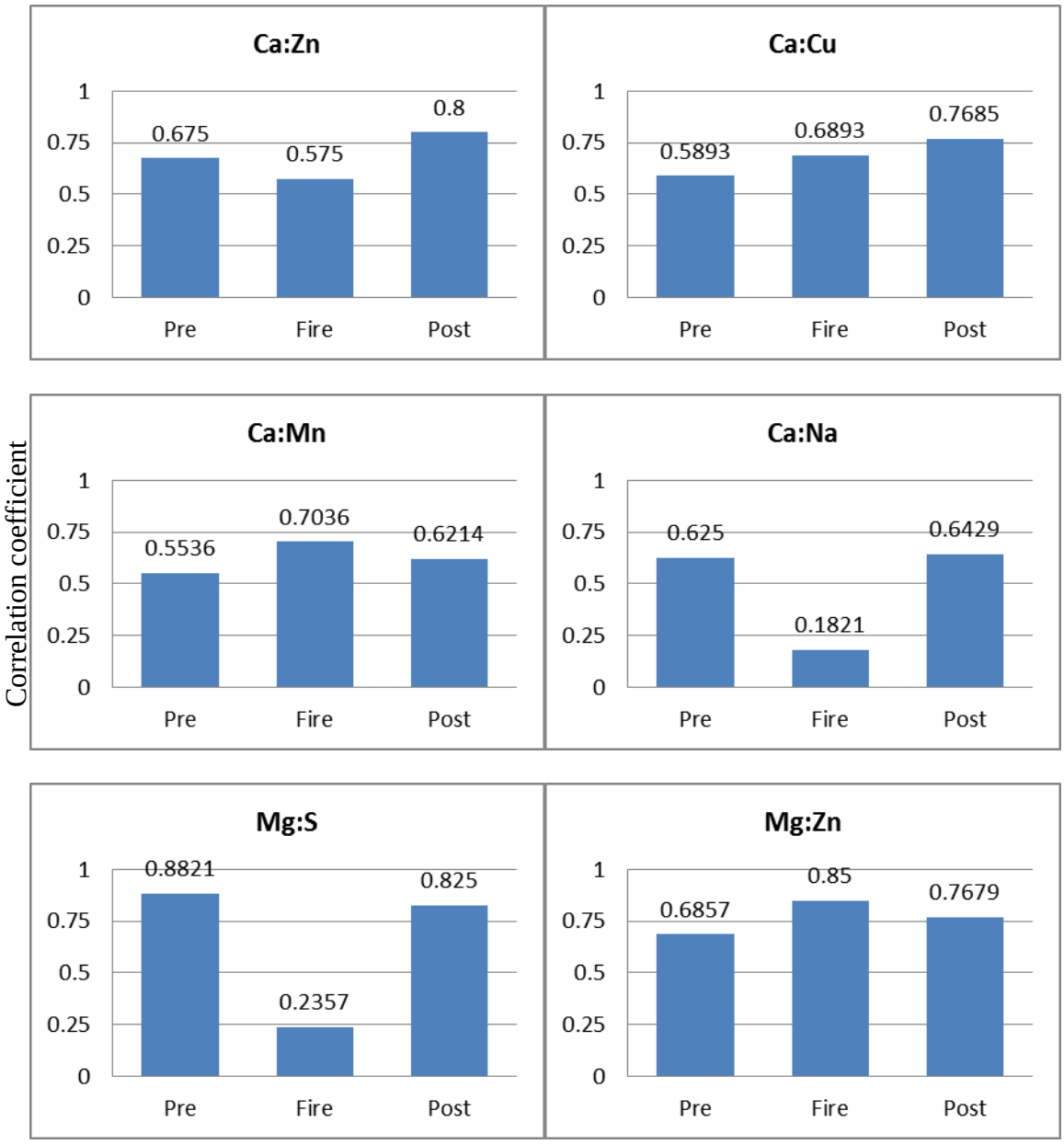

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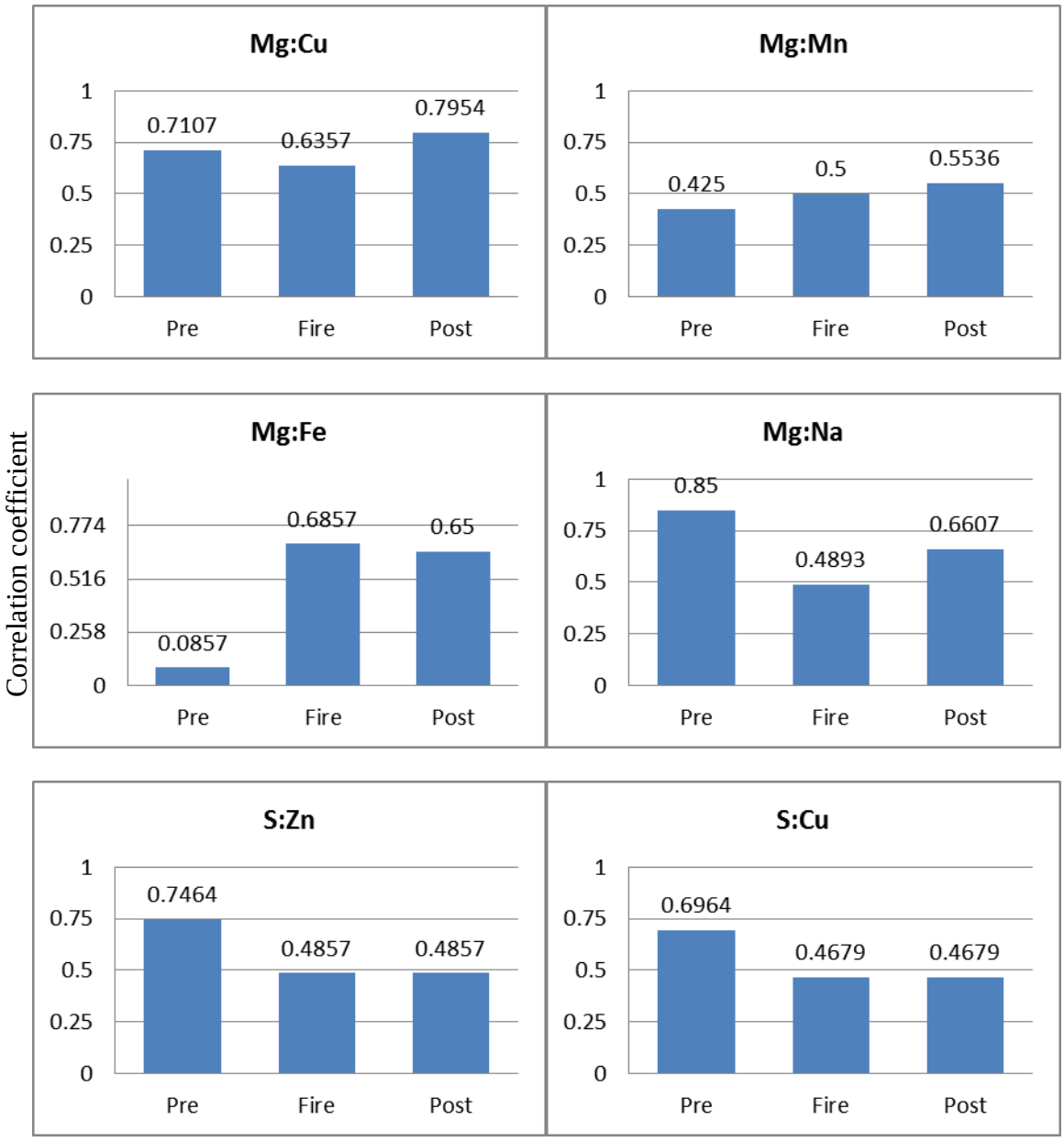

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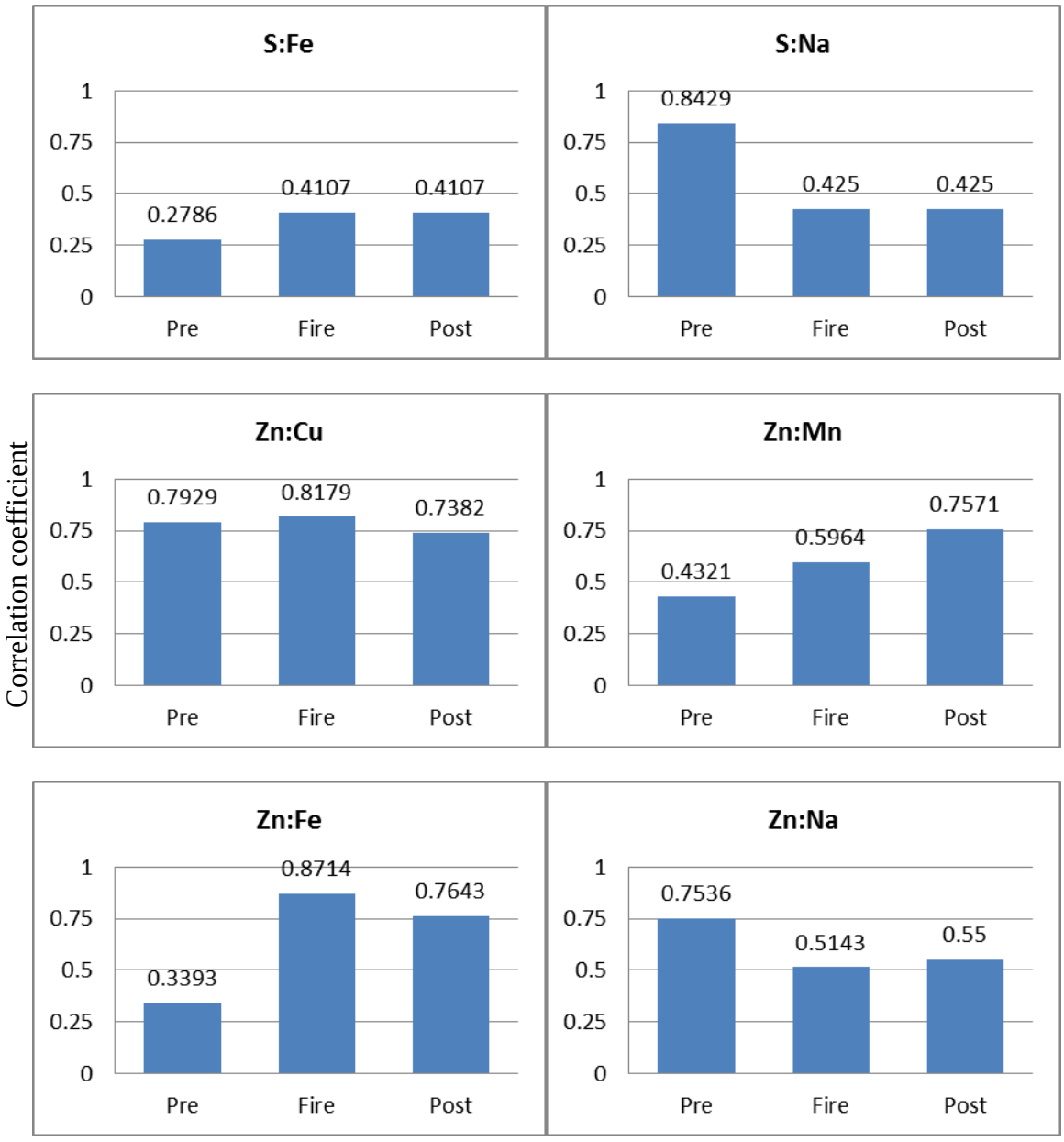

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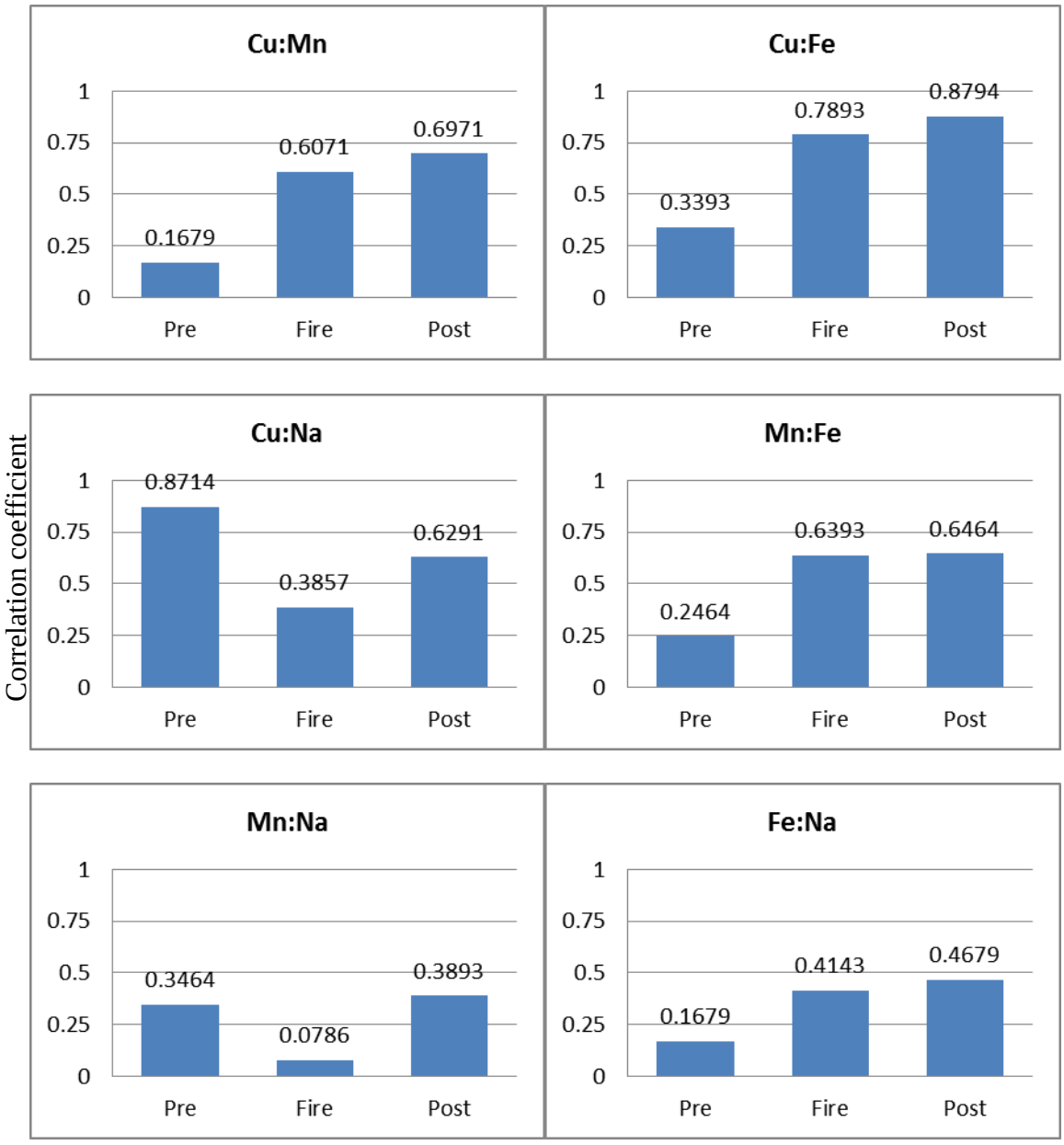

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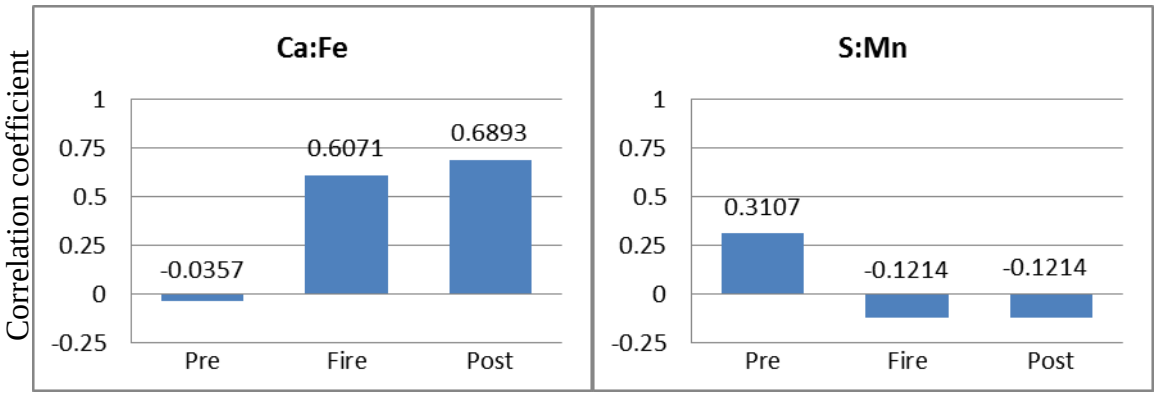

VITA

Becki Stratton was born in Philadelphia, Pennsylvania on November 10, 1980 to parents Vincent and Judith Stratton. She grew up in Marlton, New Jersey, spending her days playing in a small patch of woods where she learned to love the outdoors. She graduated from Cherokee High School in 1999 where her fascination and passion for ecology was fostered by her two favorite teachers, Mrs. Allen and Mr. Procida. Becki then earned a Bachelor of Science degree in Forestry with a minor in Wildlife and Fisheries Science from the University of Tennessee, Knoxville in 2004. While working in fire management for the U.S. Fish and Wildlife Service in Mississippi under the guidance of Tony Wilder, she became passionate about fire ecology. She received her Master of Science degree in Forestry with a minor in Statistics in August 2007 examining changes in stand structure, habitat, and small mammal population dynamics in Tennessee oak barrens under frequent fire regimes. She looks forward to researching all aspects of forest ecology in the future.